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### **ABSTRACT**

Biomass feedstocks (e.g., plant biomass, animal biomass, and municipal waste biomass) are processed to produce useful products, such as fuels. For example, systems are described that can be useful for separating solids from liquids of saccharified biomass material slurries. Many potential lignocellulosic feedstocks are available today, including agricultural residues, woody biomass, municipal waste, oil seeds/cakes and seaweed, to name a few.

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## **FILTRATION**

### **CROSS REFERENCE TO RELATED APPLICATIONS**

5           This application claims priority from the following provisional applications:  
USSN 61/774,684, filed March 8, 2013; USSN 61/774,773, filed March 8, 2013; USSN  
61/774,731, filed March 8, 2013; USSN 61/774,735, filed March 8, 2013; USSN  
61/774,740, filed March 8, 2013; USSN 61/774,744, filed March 8, 2013; USSN  
61/774,746, filed March 8, 2013; USSN 61/774,750, filed March 8, 2013; USSN  
10 61/774,752, filed March 8, 2013; USSN 61/774,754, filed March 8, 2013; USSN  
61/774,775, filed March 8, 2013; USSN 61/774,780, filed March 8, 2013; USSN  
61/774,761, filed March 8, 2013; USSN 61/774,723, filed March 8, 2013; and USSN  
61/793,336, filed March 15, 2013. The full disclosure of each of these provisional  
applications is incorporated by reference herein.

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### **BACKGROUND**

Many potential lignocellulosic feedstocks are available today, including  
agricultural residues, woody biomass, municipal waste, oilseeds/cakes and seaweed, to  
20 name a few. At present, these materials are often under-utilized, being used, for  
example, as animal feed, biocompost materials, burned in a co-generation facility or even  
landfilled.

Lignocellulosic biomass includes crystalline cellulose fibrils embedded in a  
hemicellulose matrix, surrounded by lignin. This produces a compact matrix that is  
25 difficult to access by enzymes and other chemical, biochemical and/or biological  
processes. Cellulosic biomass materials (*e.g.*, biomass material from which the lignin  
has been removed) is more accessible to enzymes and other conversion processes, but

even so, naturally-occurring cellulosic materials often have low yields (relative to theoretical yields) when contacted with hydrolyzing enzymes. Lignocellulosic biomass is even more recalcitrant to enzyme attack. Furthermore, each type of lignocellulosic biomass has its own specific composition of cellulose, hemicellulose and lignin.

5

### SUMMARY

In general, the filtering of materials, e.g., biomass materials are disclosed herein. Processes are disclosed herein for saccharifying or liquifying a biomass material, e.g.,  
10 cellulose, lignocellulosic and/or starchy feedstocks, by converting biomass material to low molecular weight sugars, e.g., saccharifying the feedstock, e.g., using an enzyme, e.g., one or more cellulase and/or amylase. The invention also relates to converting a feedstock to a product, e.g., by bioprocessing, such as fermentation or other processing, such as hydrogenation or esterification. The processes include utilizing filtration to  
15 remove solids before, during or after saccharification and/or fermentation. The solids can then be, for example, used for energy cogeneration, used as a fermentation additive (e.g., nutrient), or used as another feed material.

Methods, such as the saccharification of biomass to produce sugars, produce liquids that can be viscous due to various oligomers and the high loading of solids. In  
20 order to further process the materials, e.g., sugars or the solids in the slurries themselves, it is often advantageous to separate the liquids from the solids. Methods that involve dilution (e.g., with water) can be utilized to aid in processing, but these methods can incur a downstream cost associated with the removal of added diluents. Some of the methods described herein allow for the filtration of these highly loaded and viscous feed-  
25 streams without clogging and/or without significant dilution.

Generally the invention features systems and methods for separating solids from liquids of a slurry (e.g., containing solids and dissolved solids suspended in a liquid) including applying a saccharified biomass material slurry to a surface (e.g., to an outer  
portion) of a filter device. Optionally, a vibratory screener can be utilized to remove  
30 some of the solids prior to applying the saccharified material to the surface (e.g., outer portion) of the filter device. For example, the filter device can be a rotary drum filter

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device (e.g., a rotary vacuum drum filter device). Optionally the saccharified biomass material slurry comprises saccharified sugars, such as sugars selected from the group consisting of glucose, xylose and mixtures of these. For example, the sugars can be dissolved in and/or suspended in a solvent, such as water and/or a non-aqueous solvent.

5 The saccharified material can also include cells, such as cells selected from the group consisting of yeast cells, bacterial cells, fungal cells and mixture of these cells. Optionally the saccharified material can include protein material, such as enzyme material, denatured protein material, peptides, peptide residues, amino acids and/or denatured enzyme material. The saccharified material can include an acid (e.g., lactic  
10 acid, butyric acid and/or acetic acid) and/or an alcohol (e.g., ethanol and/or butanol). The saccharified material can include fermentation products. In some instances, the saccharified material is devoid of one or more soluble sugars from which the biomass is composed. For example, the saccharified material can be devoid of, glucose and/or xylose, such where the sugar has been removed by any means (e.g., selective fermentation  
15 of one or more of the sugars, chemical separation and removal of one or more of the sugars).

In some implementations, the biomass has been saccharified using one or more saccharification agents. Optionally, the saccharification agent is selected from the group consisting of enzymes, acids, bases, oxidants and mixtures of these. Optionally, the  
20 selected saccharification agents can be combined in any order to saccharify the biomass, for example, the biomass can be treated with an acid and then with an enzyme, or with an oxidant and then with an enzyme. In some implementations, the saccharification agent includes sulfuric acid and an enzyme. Optionally or additionally, the saccharification agent is a cellulolytic enzyme.

25 In some implementations, the biomass has been treated to reduce its recalcitrance prior to saccharification. For example, the recalcitrance of the biomass can be reduced relative to biomass (e.g., biomass feedstock) in its native state prior to saccharification. In some instances, reducing the recalcitrance of the feedstock includes treating the feedstock with a physical treatment. The treatment can include, for example, irradiation  
30 (e.g., electron beam radiation), sonication, pyrolysis, oxidation, steam explosion,

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chemical treatment, mechanical treatment and combinations of these treatments. The treatment can include the application of any one or more of the treatments disclosed herein, applied alone or in any desired combination, and applied once or multiple times.

Optionally, the methods described herein utilize a filter device e.g., a rotary drum  
5 filter device, that includes a rotatable drum having a filter face through which liquid of the saccharified material (e.g., liquids of the slurry) can pass, while retaining solids of the saccharified material thereon. The filter face can have a filter aid extending outwardly therefrom. Optionally the filter aid covers substantially the entire filter face of the rotatable drum. The filter aid can include, for example, a filter aid selected from the  
10 group consisting of diatomaceous earth, celite, silica, pumice, perlite, alumina, zeolites, sand, cellulosic material, (e.g. SOLKA-FLOC®, International Fiber Corporation, North Tonawanda, NY) lignocellulosic material, and mixtures of these. Optionally, the filter aid extends from the filter face a distance of from about 0.5 mm to about 250 mm, such as between about 1 mm and about 100 mm or about 1 mm to about 50 mm or between  
15 about 2 mm to about 25 mm. The rotatable drum includes an inner portion wherein the inner portion is maintained at a lower pressure than the filter face. For example, the pressure difference between the inner portion and the filter face can be maintained between about 20 and about 25 inches of Hg while separating solids from liquids in the slurry (e.g., the saccharified biomass). Optionally, the inner portion of the drum is in  
20 communication with a vacuum pump and/or a vacuum source, e.g., for maintaining the pressure as described above. Optionally, the filter face of the filter device includes a filter cloth such as a woven cloth, e.g., having a weave selected from the group consisting of a twill weave, a plain weave, a satin weave, a knot weave, a basket weave, an oxford weave and combinations of these. Optionally, the filter cloth has a porosity  
25 rating ranging from about 1 to 100 CFM/ft<sup>2</sup> (e.g., about 1-3, about 1-10, about 10-30, about 15-20, about 30-50, about 30-40, about 50 to 70). Optionally, the filter device includes a knife for continuously removing the solids deposited on the drum face as the drum is rotated relative to the knife. For example, the knife moves in a direction perpendicularly towards the drum face at a rate adjusted to continuously removes an  
30 interfacial region comprising the solids of the saccharified material and the filter aid. The rate, for example, can be adjusted to maintain a preset pressure difference between the

inner and outer portions of the drum. Filtering biomass materials that have been processed, e.g., saccharified and/or fermented, can be challenging and slow. The processed biomass includes particles of various sizes and shapes (e.g., fibers, granular particles, micro-particles, nano-particles, colloids and larger particles), polymers (e.g., enzymes, proteins, polysaccharides, lignin), live and/or dead cells (e.g., from yeast, 5 bacteria or fungi used to process the biomass), and small molecules (e.g., amino acids, monomeric sugars, organic acids, alcohols). The biomass filtering systems described herein generally resist clogging and allow for clarification of even the thickest biomass slurries.

10 Implementations of the invention can optionally include one or more of the following summarized features. In some implementations, the selected features can be applied or utilized in any order while in other implementations a specific selected sequence is applied or utilized. Individual features can be applied or utilized more than once in any sequence and even continuously. In addition, an entire sequence, or a portion 15 of a sequence, of applied or utilized features can be applied or utilized once, repeatedly or continuously in any order. In some optional implementations, the features can be applied or utilized with different, or where applicable the same, set or varied, quantitative or qualitative parameters as determined by a person skilled in the art. For example, parameters of the features such as size, individual dimensions (e.g., length, width, 20 height), location of, degree (e.g., to what extent such as the degree of recalcitrance), duration, frequency of use, density, concentration, intensity and speed can be varied or set, where applicable as determined by a person of skill in the art.

Features, for example, include: a method of separating solids from liquids of a slurry; saccharifying a biomass and applying the saccharified biomass to a surface of a 25 filter device; a method of separating solids from liquids of a slurry; saccharifying a biomass and applying the saccharified biomass to an outer portion of a filter device; utilizing a rotary drum filter device; filtering a saccharified biomass comprising glucose; filtering a saccharified biomass comprising xylose; filtering a saccharified biomass comprising an aqueous solvent; filtering a saccharified biomass comprising a non- 30 aqueous solvent; filtering a saccharified biomass that includes cells; filtering a

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saccharified biomass that includes yeast cells; filtering a saccharified biomass that includes bacterial cells; filtering a saccharified biomass that includes fungal cells; filtering a saccharified biomass that includes protein material; filtering a saccharified biomass that includes substantially denatured protein material; filtering a saccharified biomass that includes protein material; filtering a saccharified biomass that includes enzyme material; filtering a saccharified biomass that includes a fermentation product; filtering a saccharified biomass that includes an alcohol; filtering a saccharified biomass that includes ethanol; filtering a saccharified biomass that includes butanol; filtering a saccharified biomass that includes an organic acid; filtering a saccharified biomass that includes butyric acid; filtering a saccharified biomass that is substantially devoid of one or more soluble sugars from which the biomass is composed; utilizing a biomass composition that includes glucose and filtering the saccharified biomass when it is substantially devoid of glucose; utilizing a biomass composition that includes xylose and filtering the saccharified biomass when it is substantially devoid of xylose; utilizing a biomass that has been saccharified using one or more saccharification agent; utilizing a biomass that has been saccharified by a saccharification agent that includes enzymes; utilizing a biomass has been saccharified by a saccharification agent that includes acids; utilizing a biomass that has been saccharified by a saccharification agent that includes bases; utilizing a biomass that has been saccharified by a saccharification agent that includes oxidants; utilizing a saccharification agent that includes sulfuric acid; utilizing a saccharification agent that includes a cellulolytic enzyme; utilizing a biomass that has been treated to reduce its recalcitrance prior to saccharification; reducing the recalcitrance of a biomass by treating the biomass with electron beam irradiation prior to saccharification; utilizing a filter device that comprises a rotatable drum having a filter face through which liquid of a saccharified material can pass, while retaining solids of the saccharified material thereon; utilizing a filter device that comprises a rotatable drum having a filter face and the filter face has a filter aid extending outwardly therefrom; utilizing a filter aid that covers substantially the entire filter face of a rotatable drum filter device; utilizing a filter aid that includes diatomaceous earth; utilizing celite on a filter face; utilizing diatomaceous earth on a filter face; utilizing pumice on a filter face; utilizing perlite on a filter face; utilizing alumina on a filter face; utilizing zeolites on a



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filter face; utilizing sand on a filter face; utilizing cellulosic material on a filter face; utilizing lignocellulosic material on a filter face; a filter aid that extends from a filter face a distance of from between about 0.5 mm to about 250 mm; a filter aid that extends from a filter face a distance of from between about 1 mm and about 100 mm; a filter aid that extends from a filter face a distance of from between about 1 mm to about 50 mm; a filter aid that extends from the filter face a distance of from between about 0.5 mm to about 250 mm; a filter aid that extends from the filter face a distance of from between about 2 mm to about 25 mm; a filter device that includes a rotatable drum with an inner portion and the inner portion is maintained at a lower pressure than the filter face; utilizing a filter device that includes a rotatable drum with an inner portion and the pressure difference between the inner portion and the filter face is maintained between about 20 and about 25 inches of Hg; utilizing a filter device that includes a rotatable drum with an inner portion and the inner portion is in communication with a vacuum pump; utilizing a filter device that includes a rotatable drum with an inner portion and the inner portion is in communication with a vacuum source; utilizing a filter device that includes a filter cloth; utilizing a filter device that includes a woven filter cloth; utilizing a filter device that includes a twill weave woven filter cloth; utilizing a filter device that includes a plain weave woven filter cloth; utilizing a filter device that includes a satin weave woven filter cloth; utilizing a filter device that includes a knot weave woven filter cloth; utilizing a filter device that includes a basket weave woven filter cloth; utilizing a filter device that includes an oxford weave woven filter cloth; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 1 to 100 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 1 to 3 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 1 to 10 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 10 to 30 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 15 to 20 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 30 to 50 CFM/ft<sup>2</sup>; utilizing a filter device that includes a filter cloth that has a porosity rating range from about 50 to 70 CFM/ft<sup>2</sup>; utilizing a filter device that comprises a rotatable drum having a filter face and a knife for continuously removing the

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solids deposited on the drum face as the drum is rotated relative to the knife; utilizing a filter device that comprises a rotatable drum having a filter face and a knife for continuously removing the solids deposited on the drum face as the drum is rotated relative to the knife and the knife moves in a direction perpendicularly towards the drum face at a rate adjusted to continuously removes an interfacial region comprising the solids of a saccharified material and a filter aid; utilizing a vibratory screener to remove solids from the saccharified biomass prior to and/or after applying the saccharified biomass to the outer portion of the filter device.

All publications, patent applications, patents, and other references mentioned herein or attached hereto are incorporated by reference in their entirety for all that they contain.

#### **DESCRIPTION OF THE DRAWINGS**

FIG. 1 is a highly diagrammatic view of a rotary vacuum drum filtration (RVDF) system.

FIG. 1A is an enlarged view of an area of the RVDF system of FIG. 1, where solids are scraped from the drum.

FIG. 2 shows some filter cloths.

FIGS. 3A, 3B and 3C are flow diagrams illustrating examples of processes for filtration and concentration.

FIG. 4A shows a perspective exploded view of Rotary Pressure Filtration device. FIGS 4B, 4C, 4D and 4E show side diagrammatic views illustrating the method of processing materials utilizing the filtration device.

FIG. 5A shows a side diagrammatic view of Belt filter. FIG. 5B, 5C, 5D and 5E show detail views of various zones of the filtration device.

## DETAILED DESCRIPTION

Using the methods described herein, biomass (e.g., plant biomass, animal biomass, paper, and municipal waste biomass) can be processed to produce useful intermediates and products such as organic acids, salts of organic acids, anhydrides, esters of organic acids and fuels, e.g., fuels for internal combustion engines or feedstocks for fuel cells.

Many of the methods disclosed herein involve saccharification of biomass to produce sugars, and in some cases fermentation (or other bioprocessing or chemical transformations) of the resulting sugars to form other products and/or intermediates. After saccharification and/or fermentation (or other processing), it is often desirable to remove solids by a filtration process and in some cases to concentrate the liquid filtrate. The present disclosure pertains to techniques for accomplishing this filtration generally without clogging even with a thick (or highly loaded slurries).

FIG. 1 shows an example of a rotary vacuum drum filtration (RVDF) system **10**. RVDF system **10** includes a drum **12**, the lower portion of which is positioned in a tank **13**. The interior of the drum is placed under vacuum, e.g., by communication with vacuum pump **34**. A moisture trap or water air separator **32** may be provided to reduce moisture drawn into the vacuum pump. In some implementations, the drum includes channels that extend from the surface of the drum to a central hollow axis (not shown), and the outer surface of the drum is covered by a filter cloth (also not shown). The filter cloth, when the drum is in use, forms the outer surface of drum **12**. Some details of the filter cloth will be discussed below, referring to FIG. 2, after some more details of FIG. 1 are discussed.

Due to the porosity of the filter cloth and the underlying channels in the surface of the drum, the vacuum drawn on the interior of the drum is applied to a material on the surface of the filter cloth. Tank **13** has an inlet **14** through which filter aid, slurry or saccharified material can be fed. In some embodiments there can be separate inlets for the filter aid and saccharified material. The filter aid slurry or saccharified material can be fed from a slurry container and saccharified material container (not shown). The containers can be, for example, a drum, a tank, a fermenter, or a saccharifier e.g., that is

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100 – 150,000 gal in volume, such as 10,000 – 75,000 gal (described below). The container for the filter aid can include an agitator, such as a mixer equipped with a mixing propeller to aid in suspending the filter aid in the liquid phase (water). The container for the saccharified material and tank **13** can also include an agitator. Tank **13**  
5 is fed by a pump (e.g., positive displacement pump) or by gravity. The tank also includes an overflow outlet **16** through which material can exit the tank in case of overfilling.

When in use for filtering biomass material, such as a saccharified material, the drum is preferably initially coated with a layer **17** of filter aid. The drum is coated by  
10 adding the filter aid slurry to tank **13** and rotating drum **12** at a constant rate through the slurry while placing the interior of the drum under vacuum, for example 10-15 inches Hg. Water is drawn through the filter cloth and the filter aid is deposited as a uniform layer on this cloth. The thickness of the layer of filter aid can be varied and optimized for the process of interest (e.g., depending on the concentration and composition of the  
15 saccharified material to be processed). For example a layer **17** of filter aid slurry having a thickness between about 0.1 and 30 inches can be used ( e.g., 0.5 and 20 inches, 1 and 20 inches, 5 and 20 inches, 0.5 and 10, 0.5 to 5, 1 to 5, or 1 to 3 inches).

The filter aid increases the surface area of the drum presented to the material to be filtered and improves the filtering ability and capacity of the RVDF, generally  
20 allowing more material and/or smaller particles to be filtered. Filter aids can include diatomaceous earth, such as celite, a glass, such as a silica glass, such as volcanic amorphous glass, such as perlite, a cellulosic or lignocellulosic material, silica, alumina, zeolite, sand or mixtures of any of these filter aids.

Once the drum has been coated with filter aid, saccharified material **15** is added  
25 to tank **13**. This can be done by first exchanging the filter aid with water and then adding the saccharified material, or by simply adding the saccharified material and inter-mixing it with the filter aid slurry which is slowly diluted and replaced as the liquids are drawn through the drum filter. Alternatively, filter aid can be continuously added and the filter aid kept at a low concentration in the saccharified material, which can increase the  
30 efficiency of the filtration but which also tends to increase the cost of filtration.

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The vacuum drawn is typically increased when filtering saccharified material, for example to between 20 and 30 inches of Hg (e.g., between about 20 and 25 inches or between about 25 and 30 inches). It is preferable not to allow the surface of the drum with the filter aid and saccharified material to completely dry out, since this can lead to channel formation through the filter aid which may reduce filter efficiency. During use for filtering the saccharified material, the drum **12** is continuously rotated in the direction of arrow **A**, picking up a layer **18** of the saccharified material from the tank **13**. Optionally, the filter drum is passed beneath one or more shower heads or bars **20**, which spray a wash liquid (e.g., water) **22** onto the layer **18**. The wash liquid aids in extracting out soluble material from the solids.

The vacuum generated by vacuum pump **34** draws liquid out of the layer and into the inside of the drum **12** through the pores of the filter cloth. A filtrate receiver **30** is connected by a tube to a seal tank **26**, which receives the filtrates (wash water and liquid and dilute solids from the saccharified material). A filtrate pump **31** is engaged to move the filtrates from the system to a collection tank or other receiving area (not shown). In an alternate embodiment, the filtrate receiver is directly connected to pump **31** and the seal tank **26** is not used.

Vacuum pump **34** can be replaced by alternative vacuum sources. For example, an injector (e.g., steam injector, educator-jet pump) can be used.

Between layers **17** and **18** there is an interfacial region **40**, as shown in FIG. 1A. The interfacial region includes both saccharified material and filter aid. The interfacial region can include a concentration gradient of material, for example, perpendicular towards the drum surface, transitioning from substantially saccharified material solids to filter aid. Knife **28** removes a layer of solid from the surface of the drum, and in ideal operation, removes the saccharified material and the interfacial region. If the interfacial region is not removed, this interfacial region grows and can impede the flow of fluids through the filter drum. This impediment can cause foaming due to a pressure drop on the inside of the drum, or even complete blockage of the filtration. Thus, if the interfacial region and filter aid layer is not removed, the amount of material that can be filtered is reduced and filter aid material is wasted.

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The knife **28** is slowly moved towards the drum at a rate to continuously remove the saccharified material and the interfacial region. The rate of movement of the knife can be adjusted manually or can be adjusted automatically, and may be based on, for example, the vacuum measured in the interior of the drum, or an optical detector directed  
5 at layers **17**, **18** and/or **40**. For example, if vacuum measurement is used to control the rate of knife movement, an optimal vacuum of 20-25 inches Hg can be maintained by increasing the knife speed if the vacuum rises above 25 inches Hg, and reducing the knife speed if the vacuum drops to below 20 inches of Hg. The solution of saccharified material that is filtered can have between about 1 and 90 wt.% suspended solids (e.g.,  
10 between about 1 and 80 wt.%, between about 1 and 70 wt.%, between about 1 and 60 wt.%, between about 1 and 50 wt.%, between about 1 and 40 wt.%, between about 1 and 30 wt.%, between about 1 and 20 wt.%, between about 5 and 80 wt.%, between about 5 and 60 wt.%, between about 5 and 40 wt.%, between about 5 and 20 wt.%, between  
15 between about 10 and 80 wt.%, between about 10 and 60 wt.%, between about 10 and 40 wt.%, between about 10 and 20 wt.%, between about 15 and 80 wt.%, between about 15 and 60 wt.%, between about 15 and 40 wt.%, between about 15 and 20 wt.%, between about 20  
and 80 wt.%, between about 20 and 60 wt.%, between about 20 and 40 wt.%, between about 5 and 20 wt.%, between about 30 and 80 wt.%, between about 30 and 60 wt.%, between about 30 and 40 wt.%, between about 40 and 80 wt.%, between about 40 and 60  
20 wt.%).

As the layer **18** travels as indicated by arrow **A**, it becomes drier and drier due to the vacuum drawing liquid out of the layer and into the drum. By the time it reaches knife **28** it is relatively dry, e.g., having a moisture content of less than about 50% (less than 40%, less than 35%, less than 30%, less than 25% or even less than 20%), for  
25 example between 20 and 50%, between 20 and 40%, or between 30 and 50%.

The choice of the filter cloth depends on the application, e.g., the degree of saccharification and initial particle size of the biomass. The filter cloth is porous (e.g., permeable), to allow fluid to be drawn from material on its surface to the interior of the drum by the vacuum. For example, the filter cloth may be in the form of a wire screen,  
30 mesh, woven cloth or the like, and may be made of metal, synthetic fiber (e.g., polypropylene, polyester, polyamide, poly vinyl alcohol), natural fiber (e.g., cotton) or

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combinations of these and/or other materials. Cloths with porosity rating ranges from about 1 to 100 CFM/ft<sup>2</sup> can be utilized (e.g., about 1-3, about 1-10, about 10-30, about 15-20, about 30 to 50, about 30-40, about 50 to 70). The porosity rating, CFM/ft<sup>2</sup>, for the cloth is determined by flowing air through the cloth and is the cubic feet per minute  
5 of air passing through one square foot of the media at 0.5 inches (water column) loss. The filter cloth can be woven, for example, with a twill weave, a plain weave, a satin weave, a knot weave, a basket weave, oxford weave and combinations of these. Different filament types can also be utilized, for example, spun, multifilament, monofilament, calendared multifilament, and combinations of these. FIG. 2 shows some types of filter  
10 cloths that can be used on the drum **12**. For example **210** is a polyester plain weave filter cloth with a porosity of 45 Lm<sup>2</sup>sec<sup>-1</sup> (228.60 CFM/ ft<sup>2</sup>), **220** is a polyester twill weave with a porosity of 54 Lm<sup>2</sup>sec<sup>-1</sup> (274.32 CFM/ ft<sup>2</sup>), **230** is a polyester satin weave with a porosity of 22 Lm<sup>2</sup>sec<sup>-1</sup> (111.76 CFM/ ft<sup>2</sup>) and **240** is a polyester with a porosity of 22 Lm<sup>2</sup>sec<sup>-1</sup> (111.76 CFM/ft<sup>2</sup>).

15 Three examples of how filtration and concentration can be used in a feedstock conversion process are shown in FIGS. 3A, 3B and 3C. In all three examples, RVDF is used to separate a distillate bottom into a solid portion and a liquid portion. After removal of the solids from the distillate bottom, the filtrate can be subjected to further processing. Other examples are recognized as inventive that have not shown in  
20 the figures. For example, processes where filtration using a RVDF is applied before fermentation are optional embodiments of the invention.

In the process shown in FIG. 3A, RVDF **340** is used to filter the distillate bottom (e.g., a concentrated mixture) that is obtained by saccharification **310**, fermentation **320**, and then distillation **330**.

25 In the process shown in FIG. 3B, RVDF **340** is used to filter a mixture that is obtained by saccharification **310**, pre-filtration with a centrifuge **350**, and then distillation **330**. The centrifuge can be, for example, a continuous scroll decanter centrifuge.

In the process shown in FIG. 3C, RVDF **340** is performed after the sequence of;  
30 saccharification **310**, pre-filtration with a vibratory screening **360**, and then distillation **330**. The vibratory screener can have, for example, a mesh of between about

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10 – 200 (e.g., between about 20-100, between about 20 and 90, between about 20 and 80, between about 20 and 70, between about 20 and 60, between about 20 and 50, between about 20 and 40, between about 20 and 30, between about 30 and 90, between about 30 and 80, between about 30 and 70, between about 30 and 60, between about 30 and 50, between about 30 and 40, between about 40 and 90, between about 40 and 80, between about 40 and 70, between about 40 and 60, between about 40 and 50, between about 50 and 90, between about 50 and 80, between about 50 and 70, between about 50 and 60, between about 60 and 90, between about 60 and 80, between about 60 and 70, between about 70 and 80, between about 70 and 90, between about 80 and 90, between about 50 and 150, between about 80-180, between about 50-110, between about 60-120, between about 40 and 110, between about 150 and 200).

In some embodiments, prior to fermentation, it is preferable to remove a portion of the solids, leaving a suspension with between about 0 and 20 wt.% solids, (e.g., between about 1 and 10 wt.%, between about 5 and 10 wt.%).

15 It may also be preferable to denature any proteins that may be present after the saccharification and/or fermentation. For example the proteins may be denatured by raising or lowering the pH and/or heating the solutions.

In each case, the solids recovered by RVDF can, for example, be burned in a co-generation process to generate energy, used as a media additive in the fermentation processes discussed herein, and/or used as feed or other products.

In some implementations, centrifugation or other filtration techniques may be used instead of or in addition to RVDF. For example, lignin and other solids may be removed at any desired stage of the process by centrifugation, e.g., using a continuous scroll decanter centrifuge.

25 Some more details and reiterations of processes for treating a feedstock that can be utilized, for example, with the embodiments already discussed above, or in other embodiments, are described in the following disclosures.



### SYSTEMS FOR TREATING A FEEDSTOCK

Filtering systems, methods and equipment (e.g., RVDF) can be applied to materials that have been processed as described above and also as described anywhere herein.

5 For example, processes for conversion of a feedstock to sugars and other products, in which the methods discuss above may be used, can include, for example, optionally physically pre-treating the feedstock, e.g., to reduce its size, before and/or after this treatment, optionally treating the feedstock to reduce its recalcitrance (e.g., by irradiation), and saccharifying the feedstock to form a sugar solution. Saccharification  
10 can be performed by mixing a dispersion of the feedstock in a liquid medium, e.g., water with an enzyme, as will be discussed in detail below. During or after saccharification, the mixture (if saccharification is to be partially or completely performed en route) or solution can be transported, e.g., by pipeline, railcar, truck or barge, to a manufacturing plant. After saccharification the solution can be filtered, for example utilizing RVDF. At  
15 the plant, the solution can be bioprocessed, e.g., fermented, to produce a desired product or intermediate, which can then be processed further, e.g., by distillation, RVDF. The individual processing steps, materials used and examples of products and intermediates that may be formed will be described in detail below

### RADIATION TREATMENT

20 The feedstock can be treated with radiation to modify its structure to reduce its recalcitrance. Such treatment can, for example, reduce the average molecular weight of the feedstock, change the crystalline structure of the feedstock, and/or increase the  
25 surface area and/or porosity of the feedstock. Radiation can be by, for example electron beam, ion beam, 100 nm to 280 nm ultraviolet (UV) light, gamma or X-ray radiation. Radiation treatments and systems for treatments are discussed in US. Patent 8,142,620 and US. Patent Application Series No. 12/417, 731, the entire disclosures of which are incorporated herein by reference.

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Each form of radiation ionizes the biomass via particular interactions, as determined by the energy of the radiation. Heavy charged particles primarily ionize matter via Coulomb scattering; furthermore, these interactions produce energetic electrons that may further ionize matter. Alpha particles are identical to the nucleus of a helium atom and are produced by the alpha decay of various radioactive nuclei, such as isotopes of bismuth, polonium, astatine, radon, francium, radium, several actinides, such as actinium, thorium, uranium, neptunium, curium, californium, americium, and plutonium. Electrons interact via Coulomb scattering and bremsstrahlung radiation produced by changes in the velocity of electrons.

When particles are utilized, they can be neutral (uncharged), positively charged or negatively charged. When charged, the charged particles can bear a single positive or negative charge, or multiple charges, e.g., one, two, three or even four or more charges. In instances in which chain scission is desired to change the molecular structure of the carbohydrate containing material, positively charged particles may be desirable, in part, due to their acidic nature. When particles are utilized, the particles can have the mass of a resting electron, or greater, e.g., 500, 1000, 1500, or 2000 or more times the mass of a resting electron. For example, the particles can have a mass of from about 1 atomic unit to about 150 atomic units, e.g., from about 1 atomic unit to about 50 atomic units, or from about 1 to about 25, e.g., 1, 2, 3, 4, 5, 10, 12 or 15 atomic units.

Gamma radiation has the advantage of a significant penetration depth into a variety of material in the sample.

In embodiments in which the irradiating is performed with electromagnetic radiation, the electromagnetic radiation can have, e.g., energy per photon (in electron volts) of greater than 102 eV, e.g., greater than  $10^3$ ,  $10^4$ ,  $10^5$ ,  $10^6$ , or even greater than  $10^7$  eV. In some embodiments, the electromagnetic radiation has energy per photon of between  $10^4$  and  $10^7$ , e.g., between  $10^5$  and  $10^6$  eV. The electromagnetic radiation can have a frequency of, e.g., greater than  $10^{16}$  Hz, greater than  $10^{17}$  Hz,  $10^{18}$ ,  $10^{19}$ ,  $10^{20}$ , or even greater than  $10^{21}$  Hz. In some embodiments, the electromagnetic radiation has a frequency of between  $10^{18}$  and  $10^{22}$  Hz, e.g., between  $10^{19}$  to  $10^{21}$  Hz.

Electron bombardment may be performed using an electron beam device that has a nominal energy of less than 10 MeV, e.g., less than 7 MeV, less than 5 MeV, or less

than 2 MeV, *e.g.*, from about 0.5 to 1.5 MeV, from about 0.8 to 1.8 MeV, or from about 0.7 to 1 MeV. In some implementations the nominal energy is about 500 to 800 keV.

The electron beam may have a relatively high total beam power (the combined beam power of all accelerating heads, or, if multiple accelerators are used, of all  
5 accelerators and all heads), *e.g.*, at least 25 kW, *e.g.*, at least 30, 40, 50, 60, 65, 70, 80, 100, 125, or 150 kW. In some cases, the power is even as high as 500 kW, 750 kW, or even 1000 kW or more. In some cases the electron beam has a beam power of 1200 kW or more, *e.g.*, 1400, 1600, 1800, or even 3000 kW.

This high total beam power is usually achieved by utilizing multiple accelerating  
10 heads. For example, the electron beam device may include two, four, or more accelerating heads. The use of multiple heads, each of which has a relatively low beam power, prevents excessive temperature rise in the material, thereby preventing burning of the material, and also increases the uniformity of the dose through the thickness of the layer of material.

15 It is generally preferred that the bed of biomass material has a relatively uniform thickness. In some embodiments the thickness is less than about 1 inch (*e.g.*, less than about 0.75 inches, less than about 0.5 inches, less than about 0.25 inches, less than about 0.1 inches, between about 0.1 and 1 inch, between about 0.2 and 0.3 inches).

It is desirable to treat the material as quickly as possible. In general, it is  
20 preferred that treatment be performed at a dose rate of greater than about 0.25 Mrad per second, *e.g.*, greater than about 0.5, 0.75, 1, 1.5, 2, 5, 7, 10, 12, 15, or even greater than about 20 Mrad per second, *e.g.*, about 0.25 to 2 Mrad per second. Higher dose rates allow a higher throughput for a target (*e.g.*, the desired) dose. Higher dose rates generally require higher line speeds, to avoid thermal decomposition of the material. In one  
25 implementation, the accelerator is set for 3 MeV, 50 mA beam current, and the line speed is 24 feet/minute, for a sample thickness of about 20 mm (*e.g.*, comminuted corn cob material with a bulk density of 0.5 g/cm<sup>3</sup>).

In some embodiments, electron bombardment is performed until the material receives a total dose of at least 0.1 Mrad, 0.25 Mrad, 1 Mrad, 5 Mrad, *e.g.*, at least 10,  
30 20, 30 or at least 40 Mrad. In some embodiments, the treatment is performed until the material receives a dose of from about 10 Mrad to about 50 Mrad, *e.g.*, from about 20

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Mrad to about 40 Mrad, or from about 25 Mrad to about 30 Mrad. In some implementations, a total dose of 25 to 35 Mrad is preferred, applied ideally over a couple of passes, *e.g.*, at 5 Mrad/pass with each pass being applied for about one second.

Cooling methods, systems and equipment can be utilized before, after, during and/or  
5 between irradiations (*e.g.*, cooled screw conveyors and cooled vibratory conveyors).

Using multiple heads as discussed above, the material can be treated in multiple passes, for example, two passes at 10 to 20 Mrad/pass, *e.g.*, 12 to 18 Mrad/pass, separated by a few seconds of cool-down, or three passes of 7 to 12 Mrad/pass, *e.g.*, 5 to 20 Mrad/pass, 10 to 40 Mrad/pass, 9 to 11 Mrad/pass. As discussed herein, treating the  
10 material with several relatively low doses, rather than one high dose, tends to prevent overheating of the material and also increases dose uniformity through the thickness of the material. In some implementations, the material is stirred or otherwise mixed during or after each pass and then smoothed into a uniform layer again before the next pass, to further enhance treatment uniformity.

15 In some embodiments, electrons are accelerated to, for example, a speed of greater than 75 percent of the speed of light, *e.g.*, greater than 85, 90, 95, or 99 percent of the speed of light.

In some embodiments, any processing described herein occurs on lignocellulosic material that remains dry as acquired or that has been dried, *e.g.*, using heat and/or  
20 reduced pressure. For example, in some embodiments, the cellulosic and/or lignocellulosic material has less than about 25 wt. % retained water, measured at 25°C and at fifty percent relative humidity (*e.g.*, less than about 20 wt.%, less than about 15 wt.%, less than about 14 wt.%, less than about 13 wt.%, less than about 12 wt.%, less than about 10 wt.%, less than about 9 wt.%, less than about 8 wt.%, less than about 7  
25 wt.%, less than about 6 wt.%, less than about 5 wt.%, less than about 4 wt.%, less than about 3 wt.%, less than about 2 wt.%, less than about 1 wt.%, or less than about 0.5 wt.%.

In some embodiments, two or more ionizing sources can be used, such as two or more electron sources. For example, samples can be treated, in any order, with a beam  
30 of electrons, followed by gamma radiation and UV light having wavelengths from about

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100 nm to about 280 nm. In some embodiments, samples are treated with three ionizing radiation sources, such as a beam of electrons, gamma radiation, and energetic UV light. The biomass is conveyed through the treatment zone where it can be bombarded with electrons.

5 It may be advantageous to repeat the treatment to more thoroughly reduce the recalcitrance of the biomass and/or further modify the biomass. In particular the process parameters can be adjusted after a first (*e.g.*, second, third, fourth or more) pass depending on the recalcitrance of the material. In some embodiments, a conveyor can be used which includes a circular system where the biomass is conveyed multiple times  
10 through the various processes described above. In some other embodiments multiple treatment devices (*e.g.*, electron beam generators) are used to treat the biomass multiple (*e.g.*, 2, 3, 4 or more) times. In yet other embodiments, a single electron beam generator may be the source of multiple beams (*e.g.*, 2, 3, 4 or more beams) that can be used for treatment of the biomass.

15 The effectiveness in changing the molecular/supramolecular structure and/or reducing the recalcitrance of the carbohydrate-containing biomass depends on the electron energy used and the dose applied, while exposure time depends on the power and dose. In some embodiments, the dose rate and total dose are adjusted so as not to destroy (*e.g.*, char or burn) the biomass material. For example, the carbohydrates should  
20 not be damaged in the processing so that they can be released from the biomass intact, *e.g.* as monomeric sugars.

In some embodiments, the treatment (with any electron source or a combination of sources) is performed until the material receives a dose of at least about 0.05 Mrad, *e.g.*, at least about 0.1, 0.25, 0.5, 0.75, 1.0, 2.5, 5.0, 7.5, 10.0, 15, 20, 25, 30, 40, 50, 60,  
25 70, 80, 90, 100, 125, 150, 175, or 200 Mrad. In some embodiments, the treatment is performed until the material receives a dose of between 0.1-100 Mrad, 1-200, 5-200, 10-200, 5-150, 50-150 Mrad, 5-100, 5-50, 5-40, 10-50, 10-75, 15-50, 20-35 Mrad.

In some embodiments, relatively low doses of radiation are utilized, *e.g.*, to increase the molecular weight of a cellulosic or lignocellulosic material (with any  
30 radiation source or a combination of sources described herein). For example, a dose of at least about 0.05 Mrad, *e.g.*, at least about 0.1 Mrad or at least about 0.25, 0.5, 0.75, 1.0,

1.5, 2.0, 2.5, 3.0, 3.5, 4.0, or at least about 5.0 Mrad. In some embodiments, the irradiation is performed until the material receives a dose of between 0.1Mrad and 2.0 Mrad, e.g., between 0.5Mrad and 4.0 Mrad or between 1.0 Mrad and 3.0 Mrad.

It also can be desirable to irradiate from multiple directions, simultaneously or sequentially, in order to achieve a desired degree of penetration of radiation into the material. For example, depending on the density and moisture content of the material, such as wood, and the type of radiation source used (e.g., gamma or electron beam), the maximum penetration of radiation into the material may be only about 0.75 inch. In such instances, a thicker section (up to 1.5 inch) can be irradiated by first irradiating the material from one side, and then turning the material over and irradiating from the other side. Irradiation from multiple directions can be particularly useful with electron beam radiation, which irradiates faster than gamma radiation but typically does not achieve as great a penetration depth.

15

### **RADIATION OPAQUE MATERIALS**

The invention can include processing the material (e.g., for some of the processing steps) in a vault and/or bunker that is constructed using radiation opaque materials. In some implementations, the radiation opaque materials are selected to be capable of shielding the components from X-rays with high energy (short wavelength), which can penetrate many materials. One important factor in designing a radiation shielding enclosure is the attenuation length of the materials used, which will determine the required thickness for a particular material, blend of materials, or layered structure. The attenuation length is the penetration distance at which the radiation is reduced to approximately  $1/e$  ( $e$  = Eulers number) times that of the incident radiation. Although virtually all materials are radiation opaque if thick enough, materials containing a high compositional percentage (e.g., density) of elements that have a high  $Z$  value (atomic number) have a shorter radiation attenuation length and thus if such materials are used a thinner, lighter shielding can be provided. Examples of high  $Z$  value materials that are used in radiation shielding are tantalum and lead. Another important parameter in

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radiation shielding is the halving distance, which is the thickness of a particular material that will reduce gamma ray intensity by 50%. As an example for X-ray radiation with an energy of 0.1 MeV the halving thickness is about 15.1 mm for concrete and about 2.7 mm for lead, while with an X-ray energy of 1 MeV the halving thickness for concrete is  
5 about 44.45 mm and for lead is about 7.9 mm. Radiation opaque materials can be materials that are thick or thin so long as they can reduce the radiation that passes through to the other side. Thus, if it is desired that a particular enclosure have a low wall thickness, e.g., for light weight or due to size constraints, the material chosen should have a sufficient Z value and/or attenuation length so that its halving length is less than  
10 or equal to the desired wall thickness of the enclosure.

In some cases, the radiation opaque material may be a layered material, for example having a layer of a higher Z value material, to provide good shielding, and a layer of a lower Z value material to provide other properties (e.g., structural integrity, impact resistance, etc.). In some cases, the layered material may be a "graded-Z"  
15 laminate, e.g., including a laminate in which the layers provide a gradient from high-Z through successively lower-Z elements. In some cases the radiation opaque materials can be interlocking blocks, for example, lead and/or concrete blocks can be supplied by NELCO Worldwide (Burlington, MA), and reconfigurable vaults can be utilized.

A radiation opaque material can reduce the radiation passing through a structure  
20 (e.g., a wall, door, ceiling, enclosure, a series of these or combinations of these) formed of the material by about at least about 10 %, (e.g., at least about 20%, at least about 30%, at least about 40%, at least about 50%, at least about 60%, at least about 70%, at least about 80%, at least about 90%, at least about 95%, at least about 96%, at least about 97%, at least about 98%, at least about 99%, at least about 99.9%, at least about 99.99%,  
25 at least about 99.999%) as compared to the incident radiation. Therefore, an enclosure made of a radiation opaque material can reduce the exposure of equipment/system/components by the same amount. Radiation opaque materials can include stainless steel, metals with Z values above 25 (e.g., lead, iron), concrete, dirt, and and combinations thereof. Radiation opaque materials can include a barrier in the  
30 direction of the incident radiation of at least about 1mm (e.g., 5 mm, 10mm, 5 cm, 10 cm, 100cm, 1m or even about 10 m).

## RADIATION SOURCES

The type of radiation determines the kinds of radiation sources used as well as the radiation devices and associated equipment. The methods, systems and equipment described herein, for example for treating materials with radiation, can utilize sources as described herein as well as any other useful source.

Sources of gamma rays include radioactive nuclei, such as isotopes of cobalt, calcium, technetium, chromium, gallium, indium, iodine, iron, krypton, samarium, selenium, sodium, thallium, and xenon.

Sources of X-rays include electron beam collision with metal targets, such as tungsten or molybdenum or alloys, or compact light sources, such as those produced commercially by Lyncean.

Alpha particles are identical to the nucleus of a helium atom and are produced by the alpha decay of various radioactive nuclei, such as isotopes of bismuth, polonium, astatine, radon, francium, radium, several actinides, such as actinium, thorium, uranium, neptunium, curium, californium, americium, and plutonium.

Sources for ultraviolet radiation include deuterium or cadmium lamps.

Sources for infrared radiation include sapphire, zinc, or selenide window ceramic lamps.

Sources for microwaves include klystrons, Slevin type RF sources, or atom beam sources that employ hydrogen, oxygen, or nitrogen gases.

Accelerators used to accelerate the particles (e.g., electrons or ions) can be DC (e.g., electrostatic DC or electrodynamic DC), RF linear, magnetic induction linear or continuous wave. For example, various irradiating devices may be used in the methods disclosed herein, including field ionization sources, electrostatic ion separators, field ionization generators, thermionic emission sources, microwave discharge ion sources, recirculating or static accelerators, dynamic linear accelerators, van de Graaff accelerators, Cockroft Walton accelerators (e.g., PELLETRON® accelerators), LINACS, Dynamitrons (e.g., DYNAMITRON® accelerators), cyclotrons, synchrotrons, betatrons, transformer-type accelerators, microtrons, plasma generators, cascade accelerators, and



folded tandem accelerators. For example, cyclotron type accelerators are available from IBA, Belgium, such as the RHODOTRON™ system, while DC type accelerators are available from RDI, now IBA Industrial, such as the DYNAMITRON®. Other suitable accelerator systems include, for example: DC insulated core transformer (ICT) type systems, available from Nissin High Voltage, Japan; S-band LINACs, available from L3-PSD (USA), Linac Systems (France), Mevex (Canada), and Mitsubishi Heavy Industries (Japan); L-band LINACs, available from Iotron Industries (Canada); and ILU-based accelerators, available from Budker Laboratories (Russia). Ions and ion accelerators are discussed in Introductory Nuclear Physics, Kenneth S. Krane, John Wiley & Sons, Inc. (1988), Krsto Prelec, FIZIKA B 6 (1997) 4, 177–206, Chu, William T., “Overview of Light-Ion Beam Therapy”, Columbus-Ohio, ICRU-IAEA Meeting, 18-20 March 2006, Iwata, Y. et al., “Alternating-Phase-Focused IH-DTL for Heavy-Ion Medical Accelerators”, Proceedings of EPAC 2006, Edinburgh, Scotland, , and Leitner, C.M. et al., “Status of the Superconducting ECR Ion Source Venus”, Proceedings of EPAC 2000, Vienna, Austria. Some particle accelerators and their uses are disclosed, for example, in U.S. Pat. No. 7,931,784 to Medoff, the complete disclosure of which is incorporated herein by reference.

Electrons may be produced by radioactive nuclei that undergo beta decay, such as isotopes of iodine, cesium, technetium, and iridium. Alternatively, an electron gun can be used as an electron source via thermionic emission and accelerated through an accelerating potential. An electron gun generates electrons, which are then accelerated through a large potential (*e.g.*, greater than about 500 thousand, greater than about 1million, greater than about 2 million, greater than about 5 million, greater than about 6 million, greater than about 7 million, greater than about 8 million, greater than about 9 million, or even greater than 10 million volts) and then scanned magnetically in the x-y plane, where the electrons are initially accelerated in the z direction down the accelerator tube and extracted through a foil window. Scanning the electron beams is useful for increasing the irradiation surface when irradiating materials, *e.g.*, a biomass, that is conveyed through the scanned beam. Scanning the electron beam also distributes the thermal load homogenously on the window and helps reduce the foil window rupture due

to local heating by the electron beam. Window foil rupture is a cause of significant down-time due to subsequent necessary repairs and re-starting the electron gun.

Various other irradiating devices may be used in the methods disclosed herein, including field ionization sources, electrostatic ion separators, field ionization generators, 5 thermionic emission sources, microwave discharge ion sources, recirculating or static accelerators, dynamic linear accelerators, van de Graaff accelerators, and folded tandem accelerators. Such devices are disclosed, for example, in U.S. Pat. No. 7,931,784 to Medoff, the complete disclosure of which is incorporated herein by reference.

A beam of electrons can be used as the radiation source. A beam of electrons has 10 the advantages of high dose rates (*e.g.*, 1, 5, or even 10 Mrad per second), high throughput, less containment, and less confinement equipment. Electron beams can also have high electrical efficiency (*e.g.*, 80%), allowing for lower energy usage relative to other radiation methods, which can translate into a lower cost of operation and lower greenhouse gas emissions corresponding to the smaller amount of energy used. Electron 15 beams can be generated, *e.g.*, by electrostatic generators, cascade generators, transformer generators, low energy accelerators with a scanning system, low energy accelerators with a linear cathode, linear accelerators, and pulsed accelerators.

Electrons can also be more efficient at causing changes in the molecular structure of carbohydrate-containing materials, for example, by the mechanism of chain scission. 20 In addition, electrons having energies of 0.5-10 MeV can penetrate low density materials, such as the biomass materials described herein, *e.g.*, materials having a bulk density of less than 0.5 g/cm<sup>3</sup>, and a depth of 0.3-10 cm. Electrons as an ionizing radiation source can be useful, *e.g.*, for relatively thin piles, layers or beds of materials, *e.g.*, less than about 0.5 inch, *e.g.*, less than about 0.4 inch, 0.3 inch, 0.25 inch, or less 25 than about 0.1 inch. In some embodiments, the energy of each electron of the electron beam is from about 0.3 MeV to about 2.0 MeV (million electron volts), *e.g.*, from about 0.5 MeV to about 1.5 MeV, or from about 0.7 MeV to about 1.25 MeV. Methods of irradiating materials are discussed in U.S. Pat. App. Pub. 2012/0100577 A1, filed October 18, 2011, the entire disclosure of which is herein incorporated by reference.

30 Electron beam irradiation devices may be procured commercially or built. For example, elements or components such inductors, capacitors, casings, power sources,

cables, wiring, voltage control systems, current control elements, insulating material, microcontrollers and cooling equipment can be purchased and assembled into a device. Optionally, a commercial device can be modified and/or adapted. For example, devices and components can be purchased from any of the commercial sources described herein

5 including Ion Beam Applications (Louvain-la-Neuve, Belgium), Wasik Associates Inc. (Dracut, MA), NHV Corporation (Japan), the Titan Corporation (San Diego, CA), Vivirad High Voltage Corp (Billerica, MA) and/or Budker Laboratories (Russia). Typical electron energies can be 0.5 MeV, 1 MeV, 2 MeV, 4.5 MeV, 7.5 MeV, or 10 MeV. Typical electron beam irradiation device power can be 1 kW, 5 kW, 10 kW, 20

10 kW, 50 kW, 60 kW, 70 kW, 80 kW, 90 kW, 100 kW, 125 kW, 150 kW, 175 kW, 200 kW, 250 kW, 300 kW, 350 kW, 400 kW, 450 kW, 500 kW, 600 kW, 700 kW, 800 kW, 900 kW or even 1000 kW. Accelerators that can be used include NHV irradiators medium energy series EPS-500 (e.g., 500 kV accelerator voltage and 65, 100 or 150 mA beam current), EPS-800 (e.g., 800 kV accelerator voltage and 65 or 100 mA beam

15 current), or EPS-1000 (e.g., 1000 kV accelerator voltage and 65 or 100 mA beam current). Also, accelerators from NHV's high energy series can be used such as EPS-1500 (e.g., 1500 kV accelerator voltage and 65 mA beam current), EPS-2000 (e.g., 2000 kV accelerator voltage and 50 mA beam current), EPS-3000 (e.g., 3000 kV accelerator voltage and 50 mA beam current) and EPS-5000 (e.g., 5000 and 30 mA beam current).

20 Tradeoffs in considering electron beam irradiation device power specifications include cost to operate, capital costs, depreciation, and device footprint. Tradeoffs in considering exposure dose levels of electron beam irradiation would be energy costs and environment, safety, and health (ESH) concerns. Typically, generators are housed in a vault, *e.g.*, of lead or concrete, especially for production from X-rays that are generated

25 in the process. Tradeoffs in considering electron energies include energy costs.

The electron beam irradiation device can produce either a fixed beam or a scanning beam. A scanning beam may be advantageous with large scan sweep length and high scan speeds, as this would effectively replace a large, fixed beam width. Further, available sweep widths of 0.5 m, 1 m, 2 m or more are available. The scanning

30 beam is preferred in most embodiments described herein because of the larger scan width and reduced possibility of local heating and failure of the windows.

## ELECTRON GUNS – WINDOWS

The extraction system for an electron accelerator can include two window foils. The cooling gas in the two foil window extraction system can be a purge gas or a mixture, for example air, or a pure gas. In one embodiment the gas is an inert gas such as nitrogen, argon, helium and or carbon dioxide. It is preferred to use a gas rather than a liquid since energy losses to the electron beam are minimized. Mixtures of pure gas can also be used, either pre-mixed or mixed in line prior to impinging on the windows or in the space between the windows. The cooling gas can be cooled, for example, by using a heat exchange system (e.g., a chiller) and/or by using boil off from a condensed gas (e.g., liquid nitrogen, liquid helium). Window foils are described in PCT/US2013/64332 filed October 10, 2013 the full disclosure of which is incorporated by reference herein

## HEATING AND THROUGHPUT DURING RADIATION TREATMENT

Several processes can occur in biomass when electrons from an electron beam interact with matter in inelastic collisions. For example, ionization of the material, chain scission of polymers in the material, cross linking of polymers in the material, oxidation of the material, generation of X-rays (“Bremsstrahlung”) and vibrational excitation of molecules (e.g., phonon generation). Without being bound to a particular mechanism, the reduction in recalcitrance can be due to several of these inelastic collision effects, for example ionization, chain scission of polymers, oxidation and phonon generation. Some of the effects (e.g., especially X-ray generation), necessitate shielding and engineering barriers, for example, enclosing the irradiation processes in a concrete (or other radiation opaque material) vault. Another effect of irradiation, vibrational excitation, is equivalent to heating up the sample. Heating the sample by irradiation can help in recalcitrance reduction, but excessive heating can destroy the material, as will be explained below.

The adiabatic temperature rise ( $\Delta T$ ) from adsorption of ionizing radiation is given by the equation:  $\Delta T = D/C_p$ : where  $D$  is the average dose in kGy,  $C_p$  is the heat capacity in J/g °C, and  $\Delta T$  is the change in temperature in °C. A typical dry biomass material will

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have a heat capacity close to 2. Wet biomass will have a higher heat capacity dependent on the amount of water since the heat capacity of water is very high ( 4.19 J/g °C). Metals have much lower heat capacities, for example, 304 stainless steel has a heat capacity of 0.5 J/g °C. The temperature change due to the instant adsorption of radiation

5 in a biomass and stainless steel for various doses of radiation is shown in Table 1. At high temperatures, deviation from the calculated temperatures is expected due to decomposition of the biomass.

Table 1: Calculated Temperature increase for biomass and stainless steel.

Dose (Mrad)	Estimated Biomass ΔT (°C)	Steel ΔT (°C)
10	50	200
50	250, Decomposition	1000
100	500, Decomposition	2000
150	750, Decomposition	3000
200	1000, Decomposition	4000

10

High temperatures can destroy and or modify the biopolymers in biomass so that the polymers (e.g., cellulose) are unsuitable for further processing. A biomass subjected to high temperatures can become dark, sticky and give off odors indicating decomposition. The stickiness can even make the material hard to convey. The odors can

15 be unpleasant and be a safety issue. In fact, keeping the biomass below about 200°C has been found to be beneficial in the processes described herein (e.g., below about 190°C, below about 180°C, below about 170°C, below about 160°C, below about 150°C, below about 140°C, below about 130°C, below about 120°C, below about 110°C, between about 60°C and 180°C, between about 60°C and 160°C, between about 60°C and 150°C,

20 between about 60°C and 140°C, between about 60°C and 130°C, between about 60°C and 120°C, between about 80°C and 180°C, between about 100°C and 180°C, between about 120°C and 180°C, between about 140°C and 180°C, between about 160°C and 180°C, between about 100°C and 140°C, between about 80°C and 120°C).

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It has been found that irradiation above about 10 Mrad is desirable for the processes described herein (e.g., reduction of recalcitrance). A high throughput is also desirable so that the irradiation does not become a bottle neck in processing the biomass. The treatment is governed by a Dose rate equation:  $M = FP/D \cdot \text{time}$ , where M is the mass of irradiated material (Kg), F is the fraction of power that is adsorbed (unit less), P is the emitted power ( $\text{kW} = \text{Voltage in MeV} \times \text{Current in mA}$ ), time is the treatment time (sec) and D is the adsorbed dose (kGy). In an exemplary process where the fraction of adsorbed power is fixed, the Power emitted is constant and a set dosage is desired, the throughput (e.g., M, the biomass processed) can be increased by increasing the irradiation time. However, increasing the irradiation time without allowing the material to cool, can excessively heat the material as exemplified by the calculations shown above. Since biomass has a low thermal conductivity (less than about  $0.1 \text{ Wm}^{-1}\text{K}^{-1}$ ), heat dissipation is slow, unlike, for example metals (greater than about  $10 \text{ Wm}^{-1}\text{K}^{-1}$ ) which can dissipate energy quickly as long as there is a heat sink to transfer the energy to.

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### **ELECTRON GUNS – BEAM STOPS**

In some embodiments the systems and methods include a beam stop (e.g., a shutter). For example, the beam stop can be used to quickly stop or reduce the irradiation of material without powering down the electron beam device. Alternatively the beam stop can be used while powering up the electron beam, e.g., the beam stop can stop the electron beam until a beam current of a desired level is achieved. The beam stop can be placed between the primary foil window and a secondary foil window. For example the beam stop can be mounted so that it is movable, that is, so that it can be moved into and out of the beam path. Even partial coverage of the beam can be used, for example, to control the dose of irradiation. The beam stop can be mounted to the floor, to a conveyor for the biomass, to a wall, to the radiation device (e.g., at the scan horn), or to any structural support. Preferably the beam stop is fixed in relation to the scan horn so that the beam can be effectively controlled by the beam stop. The beam stop can incorporate a hinge, a rail, wheels, slots, or other means allowing for its operation in moving into and out of the beam. The beam stop can be made of any material that will

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stop at least 5% of the electrons, *e.g.*, at least 10%, 20%, 30%, 40%, 50%, 60%, 70%, at least 80%, 85%, 90%, 91%, 92%, 93%, 94%, 95%, 96%, 97%, 98%, 99% or even about 100% of the electrons.

The beam stop can be made of a metal including, but not limited to, stainless steel, lead, iron, molybdenum, silver, gold, titanium, aluminum, tin, or alloys of these, or laminates (layered materials) made with such metals (*e.g.*, metal-coated ceramic, metal-coated polymer, metal-coated composite, multilayered metal materials).

The beam stop can be cooled, for example, with a cooling fluid such as an aqueous solution or a gas. The beam stop can be partially or completely hollow, for example with cavities. Interior spaces of the beam stop can be used for cooling fluids and gases. The beam stop can be of any shape, including flat, curved, round, oval, square, rectangular, beveled and wedged shapes.

The beam stop can have perforations so as to allow some electrons through, thus controlling (*e.g.*, reducing) the levels of radiation across the whole area of the window, or in specific regions of the window. The beam stop can be a mesh formed, for example, from fibers or wires. Multiple beam stops can be used, together or independently, to control the irradiation. The beam stop can be remotely controlled, *e.g.*, by radio signal or hard wired to a motor for moving the beam into or out of position.

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### **BEAM DUMPS**

The embodiments disclosed herein can also include a beam dump when utilizing a radiation treatment. A beam dump's purpose is to safely absorb a beam of charged particles. Like a beam stop, a beam dump can be used to block the beam of charged particles. However, a beam dump is much more robust than a beam stop, and is intended to block the full power of the electron beam for an extended period of time. They are often used to block the beam as the accelerator is powering up.

Beam dumps are also designed to accommodate the heat generated by such beams, and are usually made from materials such as copper, aluminum, carbon, beryllium, tungsten, or mercury. Beam dumps can be cooled, for example, using a cooling fluid that can be in thermal contact with the beam dump.

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## BIOMASS MATERIALS

Lignocellulosic materials, such as can be used in the methods and equipment described herein include, but are not limited to, wood, particle board, forestry wastes  
5 (e.g., sawdust, aspen wood, wood chips), grasses, (e.g., switchgrass, miscanthus, cord grass, reed canary grass), grain residues, (e.g., rice hulls, oat hulls, wheat chaff, barley hulls), agricultural waste (e.g., silage, canola straw, wheat straw, barley straw, oat straw, rice straw, jute, hemp, flax, bamboo, sisal, abaca, corn cobs, corn stover, soybean stover, corn fiber, alfalfa, hay, coconut hair), sugar processing residues (e.g., bagasse, beet pulp,  
10 agave bagasse), , algae, seaweed, manure, sewage, and mixtures of any of these.

In some cases, the lignocellulosic material includes corncobs. Ground or hammermilled corncobs can be spread in a layer of relatively uniform thickness for irradiation, and after irradiation are easy to disperse in the medium for further processing. To facilitate harvest and collection, in some cases the entire corn plant is  
15 used, including the corn stalk, corn kernels, and in some cases even the root system of the plant.

Advantageously, no additional nutrients (other than a nitrogen source, e.g., urea or ammonia) are required during fermentation of corncobs or cellulosic or lignocellulosic materials containing significant amounts of corncobs.

20 Corncobs, before and after comminution, are also easier to convey and disperse, and have a lesser tendency to form explosive mixtures in air than other cellulosic or lignocellulosic materials such as hay and grasses.

Cellulosic materials include, for example, paper, paper products, paper waste, paper pulp, pigmented papers, loaded papers, coated papers, filled papers, magazines,  
25 printed matter (e.g., books, catalogs, manuals, labels, calendars, greeting cards, brochures, prospectuses, newsprint), printer paper, polycoated paper, card stock, cardboard, paperboard, materials having a high  $\alpha$ -cellulose content such as cotton, and mixtures of any of these. For example paper products as described in U.S. App. No. 13/396,365 (“Magazine Feedstocks” by Medoff *et al.*, filed February 14, 2012), the full  
30 disclosure of which is incorporated herein by reference.



Cellulosic materials can also include lignocellulosic materials which have been partially or fully de-lignified.

In some instances other biomass materials can be utilized, for example starchy materials. Starchy materials include starch itself, *e.g.*, corn starch, wheat starch, potato starch or rice starch, a derivative of starch, or a material that includes starch, such as an  
5 starch or rice starch, a derivative of starch, or a material that includes starch, such as an edible food product or a crop. For example, the starchy material can be arracacha, buckwheat, banana, barley, cassava, kudzu, oca, sago, sorghum, regular household potatoes, sweet potato, taro, yams, or one or more beans, such as favas, lentils or peas. Blends of any two or more starchy materials are also starchy materials. Mixtures of  
10 starchy, cellulosic and or lignocellulosic materials can also be used. For example, a biomass can be an entire plant, a part of a plant or different parts of a plant, *e.g.*, a wheat plant, cotton plant, a corn plant, rice plant or a tree. The starchy materials can be treated by any of the methods described herein.

Microbial materials that can be used as feedstock can include, but are not limited  
15 to, any naturally occurring or genetically modified microorganism or organism that contains or is capable of providing a source of carbohydrates (*e.g.*, cellulose), for example, protists, *e.g.*, animal protists (*e.g.*, protozoa such as flagellates, amoeboids, ciliates, and sporozoa) and plant protists (*e.g.*, algae such as alveolates, chlorarachniophytes, cryptomonads, euglenids, glaucophytes, haptophytes, red algae,  
20 stramenopiles, and viridiaeplantae). Other examples include seaweed, plankton (*e.g.*, macroplankton, mesoplankton, microplankton, nanoplankton, picoplankton, and femtoplankton), phytoplankton, bacteria (*e.g.*, gram positive bacteria, gram negative bacteria, and extremophiles), yeast and/or mixtures of these. In some instances, microbial biomass can be obtained from natural sources, *e.g.*, the ocean, lakes, bodies of  
25 water, *e.g.*, salt water or fresh water, or on land. Alternatively or in addition, microbial biomass can be obtained from culture systems, *e.g.*, large scale dry and wet culture and fermentation systems.

In other embodiments, the biomass materials, such as cellulosic, starchy and lignocellulosic feedstock materials, can be obtained from transgenic microorganisms and  
30 plants that have been modified with respect to a wild type variety. Such modifications may be, for example, through the iterative steps of selection and breeding to obtain

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desired traits in a plant. Furthermore, the plants can have had genetic material removed, modified, silenced and/or added with respect to the wild type variety. For example, genetically modified plants can be produced by recombinant DNA methods, where genetic modifications include introducing or modifying specific genes from parental varieties, or, for example, by using transgenic breeding wherein a specific gene or genes are introduced to a plant from a different species of plant and/or bacteria. Another way to create genetic variation is through mutation breeding wherein new alleles are artificially created from endogenous genes. The artificial genes can be created by a variety of ways including treating the plant or seeds with, for example, chemical mutagens (*e.g.*, using alkylating agents, epoxides, alkaloids, peroxides, formaldehyde), irradiation (*e.g.*, X-rays, gamma rays, neutrons, beta particles, alpha particles, protons, deuterons, UV radiation) and temperature shocking or other external stressing and subsequent selection techniques. Other methods of providing modified genes is through error prone PCR and DNA shuffling followed by insertion of the desired modified DNA into the desired plant or seed. Methods of introducing the desired genetic variation in the seed or plant include, for example, the use of a bacterial carrier, biolistics, calcium phosphate precipitation, electroporation, gene splicing, gene silencing, lipofection, microinjection and viral carriers. Additional genetically modified materials have been described in U.S. Application Serial No 13/396,369 filed February 14, 2012 the full disclosure of which is incorporated herein by reference.

Any of the methods described herein can be practiced with mixtures of any biomass materials described herein.

### **OTHER MATERIALS**

Other materials (*e.g.*, natural or synthetic materials), for example polymers, can be treated and/or made utilizing the methods, equipment and systems described hererin. For example polyethylene (*e.g.*, linear low density ethylene and high density polyethylene), polystyrenes, sulfonated polystyrenes, poly (vinyl chloride), polyesters (*e.g.*, nylons, DACRON™, KODEL™), polyalkylene esters, poly vinyl esters, polyamides (*e.g.*, KEVLAR™), polyethylene terephthalate, cellulose acetate, acetal,

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poly acrylonitrile, polycarbonates (e.g., LEXAN™), acrylics [e.g., poly (methyl methacrylate), poly(methyl methacrylate), polyacrylnitriles], Poly urethanes, polypropylene, poly butadiene, polyisobutylene, polyacrylonitrile, polychloroprene (e.g. neoprene), poly(cis-1,4-isoprene) [e.g., natural rubber], poly(trans-1,4-isoprene) [e.g., gutta percha], phenol formaldehyde, melamine formaldehyde, epoxides, polyesters, poly amines, polycarboxylic acids, polylactic acids, polyvinyl alcohols, polyanhydrides, poly fluoro carbons (e.g., TEFLON™), silicones (e.g., silicone rubber), polysilanes, poly ethers (e.g., polyethylene oxide, polypropylene oxide), waxes, oils and mixtures of these . Also included are plastics, rubbers, elastomers, fibers, waxes, gels, oils, adhesives, thermoplastics, thermosets, biodegradable polymers, resins made with these polymers, other polymers, other materials and combinations thereof. The polymers can be made by any useful method including cationic polymerization, anionic polymerization, radical polymerization, metathesis polymerization, ring opening polymerization, graft polymerization, addition polymerization. In some cases the treatments disclosed herein can be used, for example, for radically initiated graft polymerization and cross linking. Composites of polymers, for example with glass, metals, biomass (e.g., fibers, particles), ceramics can also be treated and/or made.

Other materials that can be treated by using the methods, systems and equipment disclosed herein are ceramic materials, minerals, metals, inorganic compounds. For example, silicon and germanium crystals, silicon nitrides, metal oxides, semiconductors, insulators, cements and or conductors.

In addition, manufactured multipart or shaped materials (e.g., molded, extruded, welded, riveted, layered or combined in any way) can be treated, for example, cables, pipes, boards, enclosures, integrated semiconductor chips, circuit boards, wires, tires, windows, laminated materials, gears, belts, machines, combinations of these. For example, treating a material by the methods described herein can modify the surfaces, for example, making them susceptible to further functionalization, combinations (e.g., welding) and/or treatment can cross link the materials.

**BIOMASS MATERIAL PREPARATION – MECHANICAL TREATMENTS**

The biomass can be in a dry form, for example with less than about 35% moisture content (*e.g.*, less than about 20 %, less than about 15 %, less than about 10 % less than about 5 %, less than about 4%, less than about 3 %, less than about 2 % or even less than about 1 %). The biomass can also be delivered in a wet state, for example as a wet solid, a slurry or a suspension with at least about 10 wt% solids (*e.g.*, at least about 20 wt.%, at least about 30 wt. %, at least about 40 wt.%, at least about 50 wt.%, at least about 60 wt.%, at least about 70 wt.%).

The processes disclosed herein can utilize low bulk density materials, for example cellulosic or lignocellulosic feedstocks that have been physically pretreated to have a bulk density of less than about 0.75 g/cm<sup>3</sup>, *e.g.*, less than about 0.7, 0.65, 0.60, 0.50, 0.35, 0.25, 0.20, 0.15, 0.10, 0.05 or less, *e.g.*, less than about 0.025 g/cm<sup>3</sup>. Bulk density is determined using ASTM D1895B. Briefly, the method involves filling a measuring cylinder of known volume with a sample and obtaining a weight of the sample. The bulk density is calculated by dividing the weight of the sample in grams by the known volume of the cylinder in cubic centimeters. If desired, low bulk density materials can be densified, for example, by methods described in US. Pat. No. 7,971,809 published July 5, 2011, the entire disclosure of which is hereby incorporated by reference.

In some cases, the pre-treatment processing includes screening of the biomass material. Screening can be through a mesh or perforated plate with a desired opening size, for example, less than about 6.35 mm (1/4 inch, 0.25 inch), (*e.g.*, less than about 3.18 mm (1/8 inch, 0.125 inch), less than about 1.59 mm (1/16 inch, 0.0625 inch), is less than about 0.79 mm (1/32 inch, 0.03125 inch), *e.g.*, less than about 0.51 mm (1/50 inch, 0.02000 inch), less than about 0.40 mm (1/64 inch, 0.015625 inch), less than about 0.23 mm (0.009 inch), less than about 0.20 mm (1/128 inch, 0.0078125 inch), less than about 0.18 mm (0.007 inch), less than about 0.13 mm (0.005 inch), or even less than about 0.10 mm (1/256 inch, 0.00390625 inch)). In one configuration the desired biomass falls through the perforations or screen and thus biomass larger than the perforations or screen are not irradiated. These larger materials can be re-processed, for example by

comminuting, or they can simply be removed from processing. In another configuration material that is larger than the perforations is irradiated and the smaller material is removed by the screening process or recycled. In this kind of a configuration, the conveyor, such as a vibratory conveyor, itself (for example a part of the conveyor) can be perforated or made with a mesh. For example, in one particular embodiment the biomass  
5 material may be wet and the perforations or mesh allow water to drain away from the biomass before irradiation.

Screening of material can also be by a manual method, for example by an operator or mechanoid (*e.g.*, a robot equipped with a color, reflectivity or other sensor)  
10 that removes unwanted material. Screening can also be by magnetic screening wherein a magnet is disposed near the conveyed material and the magnetic material is removed magnetically.

Optional pre-treatment processing can include heating the material. For example a portion of a conveyor conveying the biomass or other material can be sent through a  
15 heated zone. The heated zone can be created, for example, by IR radiation, microwaves, combustion (*e.g.*, gas, coal, oil, biomass), resistive heating and/or inductive coils. The heat can be applied from at least one side or more than one side, can be continuous or periodic and can be for only a portion of the material or all the material. For example, a portion of the conveying trough can be heated by use of a heating jacket. Heating can  
20 be, for example, for the purpose of drying the material. In the case of drying the material, this can also be facilitated, with or without heating, by the movement of a gas (*e.g.*, air, oxygen, nitrogen, He, CO<sub>2</sub>, Argon) over and/or through the biomass as it is being conveyed.

Optionally, pre-treatment processing can include cooling the material. Cooling  
25 material is described in U.S. Pat. No. 7,900,857 published March 8, 2011, the disclosure of which is incorporated herein by reference. For example, cooling can be by supplying a cooling fluid, for example water (*e.g.*, with glycerol), or nitrogen (*e.g.*, liquid nitrogen) to the bottom of the conveying trough. Alternatively, a cooling gas, for example, chilled nitrogen can be blown over the biomass materials or under the conveying system.

30 Another optional pre-treatment processing method can include adding a material to the biomass or other feedstocks. The additional material can be added by, for

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example, by showering, sprinkling and or pouring the material onto the biomass as it is conveyed. Materials that can be added include, for example, metals, ceramics and/or ions as described in U.S. Pat. App. Pub. 2010/0105119 A1 (filed October 26, 2009) and U.S. Pat. App. Pub. 2010/0159569 A1 (filed December 16, 2009), the entire disclosures  
5 of which are incorporated herein by reference. Optional materials that can be added include acids and bases. Other materials that can be added are oxidants (*e.g.*, peroxides, chlorates), polymers, polymerizable monomers (*e.g.*, containing unsaturated bonds), water, catalysts, enzymes and/or organisms. Materials can be added, for example, in pure form, as a solution in a solvent (*e.g.*, water or an organic solvent) and/or as a  
10 solution. In some cases the solvent is volatile and can be made to evaporate *e.g.*, by heating and/or blowing gas as previously described. The added material may form a uniform coating on the biomass or be a homogeneous mixture of different components (*e.g.*, biomass and additional material). The added material can modulate the subsequent irradiation step by increasing the efficiency of the irradiation, damping the irradiation or  
15 changing the effect of the irradiation (*e.g.*, from electron beams to X-rays or heat). The method may have no impact on the irradiation but may be useful for further downstream processing. The added material may help in conveying the material, for example, by lowering dust levels.

Biomass can be delivered to conveyor (*e.g.*, vibratory conveyors that can be used  
20 in the vaults herein described) by a belt conveyor, a pneumatic conveyor, a screw conveyor, a hopper, a pipe, manually or by a combination of these. The biomass can, for example, be dropped, poured and/or placed onto the conveyor by any of these methods. In some embodiments the material is delivered to the conveyor using an enclosed material distribution system to help maintain a low oxygen atmosphere and/or control  
25 dust and fines. Lofted or air suspended biomass fines and dust are undesirable because these can form an explosion hazard or damage the window foils of an electron gun (if such a device is used for treating the material).

The material can be leveled to form a uniform thickness between about 0.0312 and 5 inches (*e.g.*, between about 0.0625 and 2.000 inches, between about 0.125 and 1  
30 inches, between about 0.125 and 0.5 inches, between about 0.3 and 0.9 inches, between about 0.2 and 0.5 inches between about 0.25 and 1.0 inches, between about 0.25 and 0.5

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inches, 0.100 +/- 0.025 inches, 0.150 +/- 0.025 inches, 0.200 +/- 0.025 inches, 0.250 +/-  
0.025 inches, 0.300 +/- 0.025 inches, 0.350 +/- 0.025 inches, 0.400 +/- 0.025 inches,  
0.450 +/- 0.025 inches, 0.500 +/- 0.025 inches, 0.550 +/- 0.025 inches, 0.600 +/- 0.025  
inches, 0.700 +/- 0.025 inches, 0.750 +/- 0.025 inches, 0.800 +/- 0.025 inches, 0.850 +/-  
5 0.025 inches, 0.900 +/- 0.025 inches, 0.900 +/- 0.025 inches.

Generally, it is preferred to convey the material as quickly as possible through the  
electron beam to maximize throughput. For example the material can be conveyed at  
rates of at least 1 ft/min, *e.g.*, at least 2 ft/min, at least 3 ft/min, at least 4 ft/min, at least 5  
ft/min, at least 10 ft/min, at least 15 ft/min, 20, 25, 30, 35, 40, 45, 50 ft/min. The rate of  
10 conveying is related to the beam current, for example, for a ¼ inch thick biomass and  
100 mA, the conveyor can move at about 20 ft/min to provide a useful irradiation  
dosage, at 50 mA the conveyor can move at about 10 ft/min to provide approximately the  
same irradiation dosage.

After the biomass material has been conveyed through the radiation zone,  
15 optional post-treatment processing can be done. The optional post-treatment processing  
can, for example, be a process described with respect to the pre-irradiation processing.  
For example, the biomass can be screened, heated, cooled, and/or combined with  
additives. Uniquely to post-irradiation, quenching of the radicals can occur, for example,  
quenching of radicals by the addition of fluids or gases (*e.g.*, oxygen, nitrous oxide,  
20 ammonia, liquids), using pressure, heat, and/or the addition of radical scavengers. For  
example, the biomass can be conveyed out of the enclosed conveyor and exposed to a  
gas (*e.g.*, oxygen) where it is quenched, forming caboxylated groups. In one  
embodiment the biomass is exposed during irradiation to the reactive gas or fluid.  
Quenching of biomass that has been irradiated is described in U.S. Pat. No. 8,083,906  
25 published Dec 27, 2011, the entire disclosure of which is incorporate herein by reference.

If desired, one or more mechanical treatments can be used in addition to  
irradiation to further reduce the recalcitrance of the carbohydrate-containing material.  
These processes can be applied before, during and or after irradiation.

In some cases, the mechanical treatment may include an initial preparation of the  
30 feedstock as received, *e.g.*, size reduction of materials, such as by comminution, *e.g.*,  
cutting, grinding, shearing, pulverizing or chopping. For example, in some cases, loose

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feedstock (*e.g.*, recycled paper, starchy materials, or switchgrass) is prepared by shearing or shredding. Mechanical treatment may reduce the bulk density of the carbohydrate-containing material, increase the surface area of the carbohydrate-containing material and/or decrease one or more dimensions of the carbohydrate-containing material.

5           Alternatively, or in addition, the feedstock material can be treated with another treatment, for example chemical treatments, such as with an acid (HCl, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>), a base (*e.g.*, KOH and NaOH), a chemical oxidant (*e.g.*, peroxides, chlorates, ozone), irradiation, steam explosion, pyrolysis, sonication, oxidation, chemical treatment. The treatments can be in any order and in any sequence and combinations. For example, the  
10 feedstock material can first be physically treated by one or more treatment methods, *e.g.*, chemical treatment including and in combination with acid hydrolysis (*e.g.*, utilizing HCl, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>), radiation, sonication, oxidation, pyrolysis or steam explosion, and then mechanically treated. This sequence can be advantageous since materials treated by one or more of the other treatments, *e.g.*, irradiation or pyrolysis, tend to be more brittle  
15 and, therefore, it may be easier to further change the structure of the material by mechanical treatment. As another example, a feedstock material can be conveyed through ionizing radiation using a conveyor as described herein and then mechanically treated. Chemical treatment can remove some or all of the lignin (for example, chemical pulping) and can partially or completely hydrolyze the material. The methods also can  
20 be used with pre-hydrolyzed material. The methods also can be used with material that has not been pre hydrolyzed. The methods can be used with mixtures of hydrolyzed and non-hydrolyzed materials, for example, with about 50% or more non-hydrolyzed material, with about 60% or more non- hydrolyzed material, with about 70% or more non-hydrolyzed material, with about 80% or more non-hydrolyzed material or even with  
25 90% or more non-hydrolyzed material.

In addition to size reduction, which can be performed initially and/or later in processing, mechanical treatment can also be advantageous for “opening up,” “stressing,” breaking or shattering the carbohydrate-containing materials, making the cellulose of the materials more susceptible to chain scission and/or disruption of  
30 crystalline structure during the physical treatment.



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Methods of mechanically treating the carbohydrate-containing material include, for example, milling or grinding. Milling may be performed using, for example, a hammer mill, ball mill, colloid mill, conical or cone mill, disk mill, edge mill, Wiley mill, grist mill or other mill. Grinding may be performed using, for example, a cutting/impact type grinder. Some exemplary grinders include stone grinders, pin grinders, coffee grinders, and burr grinders. Grinding or milling may be provided, for example, by a reciprocating pin or other element, as is the case in a pin mill. Other mechanical treatment methods include mechanical ripping or tearing, other methods that apply pressure to the fibers, and air attrition milling. Suitable mechanical treatments further include any other technique that continues the disruption of the internal structure of the material that was initiated by the previous processing steps.

Mechanical feed preparation systems can be configured to produce streams with specific characteristics such as, for example, specific maximum sizes, specific length-to-width, or specific surface areas ratios. Physical preparation can increase the rate of reactions, improve the movement of material on a conveyor, improve the irradiation profile of the material, improve the radiation uniformity of the material, or reduce the processing time required by opening up the materials and making them more accessible to processes and/or reagents, such as reagents in a solution.

The bulk density of feedstocks can be controlled (*e.g.*, increased). In some situations, it can be desirable to prepare a low bulk density material, *e.g.*, by densifying the material (*e.g.*, densification can make it easier and less costly to transport to another site) and then reverting the material to a lower bulk density state (*e.g.*, after transport). The material can be densified, for example from less than about 0.2 g/cc to more than about 0.9 g/cc (*e.g.*, less than about 0.3 to more than about 0.5 g/cc, less than about 0.3 to more than about 0.9 g/cc, less than about 0.5 to more than about 0.9 g/cc, less than about 0.3 to more than about 0.8 g/cc, less than about 0.2 to more than about 0.5 g/cc). For example, the material can be densified by the methods and equipment disclosed in U.S. Pat. No. 7,932,065 to Medoff and International Publication No. WO 2008/073186 (which was filed October 26, 2007, was published in English, and which designated the United States), the full disclosures of which are incorporated herein by reference. Densified materials can be processed by any of the methods described herein, or any

material processed by any of the methods described herein can be subsequently densified.

In some embodiments, the material to be processed is in the form of a fibrous material that includes fibers provided by shearing a fiber source. For example, the shearing can be performed with a rotary knife cutter.

For example, a fiber source, *e.g.*, that is recalcitrant or that has had its recalcitrance level reduced, can be sheared, *e.g.*, in a rotary knife cutter, to provide a first fibrous material. The first fibrous material is passed through a first screen, *e.g.*, having an average opening size of 1.59 mm or less (1/16 inch, 0.0625 inch), provide a second fibrous material. If desired, the fiber source can be cut prior to the shearing, *e.g.*, with a shredder. For example, when a paper is used as the fiber source, the paper can be first cut into strips that are, *e.g.*, 1/4- to 1/2-inch wide, using a shredder, *e.g.*, a counter-rotating screw shredder, such as those manufactured by Munson (Utica, N.Y.). As an alternative to shredding, the paper can be reduced in size by cutting to a desired size using a guillotine cutter. For example, the guillotine cutter can be used to cut the paper into sheets that are, *e.g.*, 10 inches wide by 12 inches long.

In some embodiments, the shearing of the fiber source and the passing of the resulting first fibrous material through a first screen are performed concurrently. The shearing and the passing can also be performed in a batch-type process.

For example, a rotary knife cutter can be used to concurrently shear the fiber source and screen the first fibrous material. A rotary knife cutter includes a hopper that can be loaded with a shredded fiber source prepared by shredding a fiber source.

In some implementations, the feedstock is physically treated prior to saccharification and/or fermentation. Physical treatment processes can include one or more of any of those described herein, such as mechanical treatment, chemical treatment, irradiation, sonication, oxidation, pyrolysis or steam explosion. Treatment methods can be used in combinations of two, three, four, or even all of these technologies (in any order). When more than one treatment method is used, the methods can be applied at the same time or at different times. Other processes that change a molecular structure of a biomass feedstock may also be used, alone or in combination with the processes disclosed herein.

Mechanical treatments that may be used, and the characteristics of the mechanically treated carbohydrate-containing materials, are described in further detail in U.S. Pat. App. Pub. 2012/0100577 A1, filed October 18, 2011, the full disclosure of which is hereby incorporated herein by reference.

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### **SONICATION, PYROLYSIS, OXIDATION, STEAM EXPLOSION**

If desired, one or more sonication, pyrolysis, oxidative, or steam explosion processes can be used instead of or in addition to irradiation to reduce or further reduce the recalcitrance of the carbohydrate-containing material. For example, these processes can be applied before, during and or after irradiation. These processes are described in detail in U.S. Pat. No. 7,932,065 to Medoff, the full disclosure of which is incorporated herein by reference.

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### **INTERMEDIATES AND PRODUCTS**

Using the processes described herein, the biomass material can be converted to one or more products, such as energy, fuels, foods and materials. For example, intermediates and products such as organic acids, salts of organic acids, anhydrides, esters of organic acids and fuels, *e.g.*, fuels for internal combustion engines or feedstocks for fuel cells can be produced. Systems and processes are described herein that can use as feedstock cellulosic and/or lignocellulosic materials that are readily available, but often can be difficult to process, *e.g.*, municipal waste streams and waste paper streams, such as streams that include newspaper, kraft paper, corrugated paper or mixtures of these.

Specific examples of products include, but are not limited to, hydrogen, sugars (*e.g.*, glucose, xylose, arabinose, mannose, galactose, fructose, disaccharides, oligosaccharides and polysaccharides), alcohols (*e.g.*, monohydric alcohols or dihydric alcohols, such as ethanol, n-propanol, isobutanol, *sec*-butanol, tert-butanol or n-butanol), hydrated or hydrous alcohols (*e.g.*, containing greater than 10%, 20%, 30% or even greater than 40% water), biodiesel, organic acids, hydrocarbons (*e.g.*, methane, ethane,

propane, isobutene, pentane, n-hexane, biodiesel, bio-gasoline and mixtures thereof), co-products (*e.g.*, proteins, such as cellulolytic proteins (enzymes) or single cell proteins), and mixtures of any of these in any combination or relative concentration, and optionally in combination with any additives (*e.g.*, fuel additives). Other examples include

5 carboxylic acids, salts of a carboxylic acid, a mixture of carboxylic acids and salts of carboxylic acids and esters of carboxylic acids (*e.g.*, methyl, ethyl and n-propyl esters), ketones (*e.g.*, acetone), aldehydes (*e.g.*, acetaldehyde), alpha and beta unsaturated acids (*e.g.*, acrylic acid) and olefins (*e.g.*, ethylene). Other alcohols and alcohol derivatives include propanol, propylene glycol, 1,4-butanediol, 1,3-propanediol, sugar alcohols (*e.g.*,

10 erythritol, glycol, glycerol, sorbitol, threitol, arabitol, ribitol, mannitol, dulcitol, fucitol, iditol, isomalt, maltitol, lactitol, xylitol and other polyols), and methyl or ethyl esters of any of these alcohols. Other products include methyl acrylate, methylmethacrylate, D-lactic acid, L-Lactic acid, pyruvic acid, polylactic acid, citric acid, formic acid, acetic acid, propionic acid, butyric acid, succinic acid, valeric acid, caproic acid, 3-

15 hydroxypropionic acid, palmitic acid, stearic acid, oxalic acid, malonic acid, glutaric acid, oleic acid, linoleic acid, glycolic acid, gamma-hydroxybutyric acid, and mixtures thereof, salts of any of these acids, mixtures of any of the acids and their respective salts.

Any combination of the above products with each other, and/or of the above products with other products, which other products may be made by the processes

20 described herein or otherwise, may be packaged together and sold as products. The products may be combined, *e.g.*, mixed, blended or co-dissolved, or may simply be packaged or sold together.

Any of the products or combinations of products described herein may be sanitized or sterilized prior to selling the products, *e.g.*, after purification or isolation or

25 even after packaging, to neutralize one or more potentially undesirable contaminants that could be present in the product(s). Such sanitation can be done with electron bombardment, for example, by at a dosage of less than about 20 Mrad, *e.g.*, from about 0.1 to 15 Mrad, from about 0.5 to 7 Mrad, or from about 1 to 3 Mrad.

The processes described herein can produce various by-product streams useful

30 for generating steam and electricity to be used in other parts of the plant (co-generation) or sold on the open market. For example, steam generated from burning by-product

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streams can be used in a distillation process. As another example, electricity generated from burning by-product streams can be used to power electron beam generators used in pretreatment.

5 The by-products used to generate steam and electricity are derived from a number of sources throughout the process. For example, anaerobic digestion of wastewater can produce a biogas high in methane and a small amount of waste biomass (sludge). As another example, post-saccharification and/or post-distillate solids (*e.g.*, unconverted lignin, cellulose, and hemicellulose remaining from the pretreatment and primary processes) can be used, *e.g.*, burned, as a fuel.

10 Other intermediates and products, including food and pharmaceutical products, are described in U.S. Pat. App. Pub. 2010/0124583 A1, published May 20, 2010, to Medoff, the full disclosure of which is hereby incorporated by reference herein.

### LIGNIN DERIVED PRODUCTS

15

The spent biomass (*e.g.*, spent lignocellulosic material) from lignocellulosic processing by the methods described are expected to have a high lignin content and in addition to being useful for producing energy through combustion in a Co-Generation plant, may have uses as other valuable products. For example, the lignin can be used as captured as a plastic, or it can be synthetically upgraded to other plastics. In some instances, it can also be converted to lignosulfonates, which can be utilized as binders, dispersants, emulsifiers or as sequestrants.

25 When used as a binder, the lignin or a lignosulfonate can, *e.g.*, be utilized in coal briquettes, in ceramics, for binding carbon black, for binding fertilizers and herbicides, as a dust suppressant, in the making of plywood and particle board, for binding animal feeds, as a binder for fiberglass, as a binder in linoleum paste and as a soil stabilizer.

When used as a dispersant, the lignin or lignosulfonates can be used, *e.g.*, concrete mixes, clay and ceramics, dyes and pigments, leather tanning and in gypsum board.

30 When used as an emulsifier, the lignin or lignosulfonates can be used, *e.g.*, in asphalt, pigments and dyes, pesticides and wax emulsions.

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When used as a sequestrant, the lignin or lignosulfonates can be used, e.g., in micro-nutrient systems, cleaning compounds and water treatment systems, e.g., for boiler and cooling systems.

For energy production lignin generally has a higher energy content than  
5 holocellulose (cellulose and hemicellulose) since it contains more carbon than  
homocellulose. For example, dry lignin can have an energy content of between about  
11,000 and 12,500 BTU per pound, compared to 7,000 and 8,000 BTU per pound of  
holocellulose. As such, lignin can be densified and converted into briquettes and pellets  
for burning. For example, the lignin can be converted into pellets by any method  
10 described herein. For a slower burning pellet or briquette, the lignin can be crosslinked,  
such as applying a radiation dose of between about 0.5 Mrad and 5 Mrad. Crosslinking  
can make a slower burning form factor. The form factor, such as a pellet or briquette,  
can be converted to a “synthetic coal” or charcoal by pyrolyzing in the absence of air,  
e.g., at between 400 and 950 °C. Prior to pyrolyzing, it can be desirable to crosslink the  
15 lignin to maintain structural integrity.

### SACCHARIFICATION

As previously described, in order to convert the feedstock to a form that can be  
20 readily processed the glucan- or xylan-containing cellulose in the feedstock can be  
hydrolyzed to low molecular weight carbohydrates, such as sugars, by a saccharifying  
agent, e.g., an enzyme or acid, a process referred to as saccharification. The low  
molecular weight carbohydrates can then be used, for example, in an existing  
manufacturing plant, such as a single cell protein plant, an enzyme manufacturing plant,  
25 or a fuel plant, e.g., an ethanol manufacturing facility.

As previously disclosed, the feedstock can be hydrolyzed using an enzyme, e.g.,  
by combining the materials and the enzyme in a solvent, e.g., in an aqueous solution.

Alternatively, the enzymes can be supplied by organisms that break down  
biomass, such as the cellulose and/or the lignin portions of the biomass, contain or  
30 manufacture various cellulolytic enzymes (cellulases), ligninases or various small

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molecule biomass-degrading metabolites. These enzymes may be a complex of enzymes that act synergistically to degrade crystalline cellulose or the lignin portions of biomass. Examples of cellulolytic enzymes include: endoglucanases, cellobiohydrolases, and cellobiases (beta-glucosidases).

5           During saccharification a cellulosic substrate can be initially hydrolyzed by endoglucanases at random locations producing oligomeric intermediates. These intermediates are then substrates for exo-splitting glucanases such as cellobiohydrolase to produce cellobiose from the ends of the cellulose polymer. Cellobiose is a water-soluble 1,4-linked dimer of glucose. Finally, cellobiase cleaves cellobiose to yield  
10           glucose. The efficiency (*e.g.*, time to hydrolyze and/or completeness of hydrolysis) of this process depends on the recalcitrance of the cellulosic material.

          Therefore, the treated biomass materials can be saccharified, by combining the material and a cellulase enzyme in a fluid medium, *e.g.*, an aqueous solution. In some cases, the material is boiled, steeped, or cooked in hot water prior to saccharification, as  
15           described in U.S. Pat. App. Pub. 2012/0100577 A1 by Medoff and Masterman, published on April 26, 2012, the entire contents of which are incorporated herein.

          The saccharification process can be partially or completely performed in a tank (*e.g.*, a tank having a volume of at least 4000, 40,000, or 500,000 L) in a manufacturing plant, and/or can be partially or completely performed in transit, *e.g.*, in a rail car, tanker  
20           truck, or in a supertanker or the hold of a ship. The time required for complete saccharification will depend on the process conditions and the carbohydrate-containing material and enzyme used. If saccharification is performed in a manufacturing plant under controlled conditions, the cellulose may be substantially entirely converted to sugar, *e.g.*, glucose in about 12-96 hours. If saccharification is performed partially or  
25           completely in transit, saccharification may take longer.

          It is generally preferred that the tank contents be mixed during saccharification, *e.g.*, using jet mixing as described in International App. No. PCT/US2010/035331, filed May 18, 2010, which was published in English as WO 2010/135380 and designated the United States, the full disclosure of which is incorporated by reference herein.

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The addition of surfactants can enhance the rate of saccharification. Examples of surfactants include non-ionic surfactants, such as a Tween® 20 or Tween® 80 polyethylene glycol surfactants, ionic surfactants, or amphoteric surfactants.

It is generally preferred that the concentration of the sugar solution resulting from saccharification be relatively high, *e.g.*, greater than 40%, or greater than 50, 60, 70, 80, 90 or even greater than 95% by weight. Water may be removed, *e.g.*, by evaporation, to increase the concentration of the sugar solution. This reduces the volume to be shipped, and also inhibits microbial growth in the solution.

Alternatively, sugar solutions of lower concentrations may be used, in which case it may be desirable to add an antimicrobial additive, *e.g.*, a broad spectrum antibiotic, in a low concentration, *e.g.*, 50 to 150 ppm. Other suitable antibiotics include amphotericin B, ampicillin, chloramphenicol, ciprofloxacin, gentamicin, hygromycin B, kanamycin, neomycin, penicillin, puromycin, streptomycin. Antibiotics will inhibit growth of microorganisms during transport and storage, and can be used at appropriate concentrations, *e.g.*, between 15 and 1000 ppm by weight, *e.g.*, between 25 and 500 ppm, or between 50 and 150 ppm. If desired, an antibiotic can be included even if the sugar concentration is relatively high. Alternatively, other additives with anti-microbial or preservative properties may be used. Preferably the antimicrobial additive(s) are food-grade.

A relatively high concentration solution can be obtained by limiting the amount of water added to the carbohydrate-containing material with the enzyme. The concentration can be controlled, *e.g.*, by controlling how much saccharification takes place. For example, concentration can be increased by adding more carbohydrate-containing material to the solution. In order to keep the sugar that is being produced in solution, a surfactant can be added, *e.g.*, one of those discussed above. Solubility can also be increased by increasing the temperature of the solution. For example, the solution can be maintained at a temperature of 40-50°C, 60-80°C, or even higher.



### SACCHARIFYING AGENTS

Suitable cellulolytic enzymes include cellulases from species in the genera *Bacillus*, *Coprinus*, *Myceliophthora*, *Cephalosporium*, *Scytalidium*, *Penicillium*,  
5 *Aspergillus*, *Pseudomonas*, *Humicola*, *Fusarium*, *Thielavia*, *Acremonium*,  
*Chrysosporium* and *Trichoderma*, especially those produced by a strain selected from the  
species *Aspergillus* (see, e.g., EP Pub. No. 0 458 162), *Humicola insolens* (reclassified as  
*Scytalidium thermophilum*, see, e.g., U.S. Pat. No. 4,435,307), *Coprinus cinereus*,  
*Fusarium oxysporum*, *Myceliophthora thermophila*, *Meripilus giganteus*, *Thielavia*  
10 *terrestris*, *Acremonium sp.* (including, but not limited to, *A. persicinum*, *A. acremonium*,  
*A. brachyphenium*, *A. dichromosporum*, *A. obclavatum*, *A. pinkertoniae*, *A. roseogriseum*,  
*A. incoloratum*, and *A. furatum*). Preferred strains include *Humicola insolens* DSM  
1800, *Fusarium oxysporum* DSM 2672, *Myceliophthora thermophila* CBS 117.65,  
*Cephalosporium sp.* RYM-202, *Acremonium sp.* CBS 478.94, *Acremonium sp.* CBS  
15 265.95, *Acremonium persicinum* CBS 169.65, *Acremonium acremonium* AHU 9519,  
*Cephalosporium sp.* CBS 535.71, *Acremonium brachyphenium* CBS 866.73, *Acremonium*  
*dichromosporum* CBS 683.73, *Acremonium obclavatum* CBS 311.74, *Acremonium*  
*pinkertoniae* CBS 157.70, *Acremonium roseogriseum* CBS 134.56, *Acremonium*  
*incoloratum* CBS 146.62, and *Acremonium furatum* CBS 299.70H. Cellulolytic  
20 enzymes may also be obtained from *Chrysosporium*, preferably a strain of  
*Chrysosporium lucknowense*. Additional strains that can be used include, but are not  
limited to, *Trichoderma* (particularly *T. viride*, *T. reesei*, and *T. koningii*), alkalophilic  
*Bacillus* (see, for example, U.S. Pat. No. 3,844,890 and EP Pub. No. 0 458 162), and  
*Streptomyces* (see, e.g., EP Pub. No. 0 458 162).

25 In addition to or in combination to enzymes, acids, bases and other chemicals  
(e.g., oxidants) can be utilized to saccharify lignocellulosic and cellulosic materials.  
These can be used in any combination or sequence (e.g., before, after and/or during  
addition of an enzyme). For example, strong mineral acids can be utilized (e.g. HCl,  
H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>) and strong bases (e.g., NaOH, KOH).

30

## SUGARS

In the processes described herein, for example, after saccharification, sugars (*e.g.*, glucose and xylose) can be isolated and/or purified. For example, sugars can be isolated and/or purified by precipitation, crystallization, chromatography (*e.g.*, simulated moving bed chromatography, high pressure chromatography), electro dialysis, centrifugation, extraction, any other isolation method known in the art, and combinations thereof.

## HYDROGENATION AND OTHER CHEMICAL TRANSFORMATIONS

The processes described herein can include hydrogenation. For example, glucose and xylose can be hydrogenated to sorbitol and xylitol respectively. Hydrogenation can be accomplished by use of a catalyst (*e.g.*, Pt/gamma-Al<sub>2</sub>O<sub>3</sub>, Ru/C, Raney Nickel, or other catalysts know in the art) in combination with H<sub>2</sub> under high pressure (*e.g.*, 10 to 12000 psi). Other types of chemical transformation of the products from the processes described herein can be used, for example, production of organic sugar derived products such (*e.g.*, furfural and furfural-derived products). Chemical transformations of sugar derived products are described in USSN 13/934,704 filed July 3, 2013, the entire disclosure of which is incorporated herein by reference in its entirety.

## FERMENTATION

Yeast and *Zymomonas* bacteria, for example, can be used for fermentation or conversion of sugar(s) to alcohol(s). Other microorganisms are discussed below. The optimum pH for fermentations is about pH 4 to 7. For example, the optimum pH for yeast is from about pH 4 to 5, while the optimum pH for *Zymomonas* is from about pH 5 to 6. Typical fermentation times are about 24 to 168 hours (*e.g.*, 24 to 96 hrs) with temperatures in the range of 20°C to 40°C (*e.g.*, 26°C to 40°C), however thermophilic microorganisms prefer higher temperatures.

In some embodiments, *e.g.*, when anaerobic organisms are used, at least a portion of the fermentation is conducted in the absence of oxygen, *e.g.*, under a blanket of an

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inert gas such as N<sub>2</sub>, Ar, He, CO<sub>2</sub> or mixtures thereof. Additionally, the mixture may have a constant purge of an inert gas flowing through the tank during part of or all of the fermentation. In some cases, anaerobic condition, can be achieved or maintained by carbon dioxide production during the fermentation and no additional inert gas is needed.

5 In some embodiments, all or a portion of the fermentation process can be interrupted before the low molecular weight sugar is completely converted to a product (*e.g.*, ethanol). The intermediate fermentation products include sugar and carbohydrates in high concentrations. The sugars and carbohydrates can be isolated via any means known in the art. These intermediate fermentation products can be used in preparation of  
10 food for human or animal consumption. Additionally or alternatively, the intermediate fermentation products can be ground to a fine particle size in a stainless-steel laboratory mill to produce a flour-like substance. Jet mixing may be used during fermentation, and in some cases saccharification and fermentation are performed in the same tank.

Nutrients for the microorganisms may be added during saccharification and/or  
15 fermentation, for example the food-based nutrient packages described in U.S. Pat. App. Pub. 2012/0052536, filed July 15, 2011, the complete disclosure of which is incorporated herein by reference.

“Fermentation” includes the methods and products that are disclosed in applications No. PCT/US2012/71093 published June 27, 2013, PCT/ US2012/71907  
20 published June 27, 2012, and PCT/US2012/71083 published June 27, 2012 the contents of which are incorporated by reference herein in their entirety.

Mobile fermenters can be utilized, as described in International App. No. PCT/US2007/074028 (which was filed July 20, 2007, was published in English as WO 2008/011598 and designated the United States) and has a U.S. issued Patent No.  
25 8,318,453, the contents of which are incorporated herein in its entirety. Similarly, the saccharification equipment can be mobile. Further, saccharification and/or fermentation may be performed in part or entirely during transit.

**FERMENTATION AGENTS**

The microorganism(s) used in fermentation can be naturally-occurring microorganisms and/or engineered microorganisms. For example, the microorganism  
 5 can be a bacterium (including, but not limited to, *e.g.*, a cellulolytic bacterium), a fungus, (including, but not limited to, *e.g.*, a yeast), a plant, a protist, *e.g.*, a protozoa or a fungus-like protest (including, but not limited to, *e.g.*, a slime mold), or an alga. When the organisms are compatible, mixtures of organisms can be utilized.

Suitable fermenting microorganisms have the ability to convert carbohydrates,  
 10 such as glucose, fructose, xylose, arabinose, mannose, galactose, oligosaccharides or polysaccharides into fermentation products. Fermenting microorganisms include strains of the genus *Sacchromyces spp.* (including, but not limited to, *S. cerevisiae* (baker's yeast), *S. distaticus*, *S. uvarum*), the genus *Kluyveromyces*, (including, but not limited to, *K. marxianus*, *K. fragilis*), the genus *Candida* (including, but not limited to, *C.*  
 15 *pseudotropicalis*, and *C. brassicae*), *Pichia stipitis* (a relative of *Candida shehatae*), the genus *Clavispora* (including, but not limited to, *C. lusitaniae* and *C. opuntiae*), the genus *Pachysolen* (including, but not limited to, *P. tannophilus*), the genus *Bretannomyces* (including, but not limited to, *e.g.*, *B. clausenii* (Philippidis, G. P., 1996, Cellulose bioconversion technology, in Handbook on Bioethanol: Production and Utilization,  
 20 Wyman, C.E., ed., Taylor & Francis, Washington, DC, 179-212)). Other suitable microorganisms include, for example, *Zymomonas mobilis*, *Clostridium spp.* (including, but not limited to, *C. thermocellum* (Philippidis, 1996, *supra*), *C.*  
*saccharobutylaceticum*, *C. tyrobutyricum* *C. saccharobutylicum*, *C. Puniceum*, *C. beijernckii*, and *C. acetobutylicum*), *Moniliella spp.* (including but not limited to *M.*  
 25 *pollinis*, *M. tomentosa*, *M. madida*, *M. nigrescens*, *M. oedocephali*, *M. megachiliensis*), *Yarrowia lipolytica*, *Aureobasidium sp.*, *Trichosporonoides sp.*, *Trigonopsis variabilis*, *Trichosporon sp.*, *Moniliellaacetoabutans sp.*, *Typhula variabilis*, *Candida magnoliae*, *Ustilaginomycetes sp.*, *Pseudozyma tsukubaensis*, yeast species of genera *Zygosaccharomyces*, *Debaryomyces*, *Hansenula* and *Pichia*, and fungi of the dematioid  
 30 genus *Torula* (*e.g.*, *T. corallina*).

Additional microorganisms include the Lactobacillus group. Examples include *Lactobacillus casei*, *Lactobacillus rhamnosus*, *Lactobacillus delbrueckii*, *Lactobacillus plantarum*, *Lactobacillus coryniformis*, e.g., *Lactobacillus coryniformis subspecies torquens*, *Lactobacillus pentosus*, *Lactobacillus brevis*. Other microorganisms include

5 *Pediococcus penosaceus*, *Rhizopus oryzae*.

Several organisms, such as bacteria, yeasts and fungi, can be utilized to ferment biomass derived products such as sugars and alcohols to succinic acid and similar products. For example, organisms can be selected from; *Actinobacillus succinogenes*, *Anaerobiospirillum succiniciproducens*, *Mannheimia succiniciproducens*, *Ruminococcus*

10 *flaverfaciens*, *Ruminococcus albus*, *Fibrobacter succinogenes*, *Bacteroides fragilis*, *Bacteroides ruminicola*, *Bacteroides amylophilus*, *Bacteriodes succinogenes*, *Mannheimia succiniciproducens*, *Corynebacterium glutamicum*, *Aspergillus niger*, *Aspergillus fumigatus*, *Byssochlamys nivea*, *Lentinus degener*, *Paecilomyces varioti*, *Penicillium viniferum*, *Saccharomyces cerevisiae*, *Enterococcus faecali*, *Prevotella*

15 *ruminicolus*, *Debaryomyces hansenii*, *Candida catenulata* VKM Y-5, *C. mycoderma* VKM Y-240, *C. rugosa* VKM Y-67, *C. paludigena* VKM Y-2443, *C. utilis* VKM Y-74, *C. utilis* 766, *C. zeylanoides* VKM Y-6, *C. zeylanoides* VKM Y-14, *C. zeylanoides* VKM Y-2324, *C. zeylanoides* VKM Y-1543, *C. zeylanoides* VKM Y-2595, *C. valida* VKM Y-934, *Kluyveromyces wickerhamii* VKM Y-589, *Pichia anomala* VKM Y-118, *P. besseyi*

20 VKM Y-2084, *P. media* VKM Y-1381, *P. guilliermondii* H-P-4, *P. guilliermondii* 916, *P. inositovora* VKM Y-2494, *Saccharomyces cerevisiae* VKM Y-381, *Torulopsis candida* 127, *T. candida* 420, *Yarrowia lipolytica* 12a, *Y. lipolytica* VKM Y-47, *Y. lipolytica* 69, *Y. lipolytica* VKM Y-57, *Y. lipolytica* 212, *Y. lipolytica* 374/4, *Y. lipolytica* 585, *Y. lipolytica* 695, *Y. lipolytica* 704, and mixtures of these organisms.

25 Many such microbial strains are publicly available, either commercially or through depositories such as the ATCC (American Type Culture Collection, Manassas, Virginia, USA), the NRRL (Agricultural Research Service Culture Collection, Peoria, Illinois, USA), or the DSMZ (Deutsche Sammlung von Mikroorganismen und Zellkulturen GmbH, Braunschweig, Germany), to name a few.

30 Commercially available yeasts include, for example, RED STAR®/Lesaffre Ethanol Red (available from Red Star/Lesaffre, USA), FALI® (available from

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Fleischmann's Yeast, a division of Burns Philip Food Inc., USA), SUPERSTART® (available from Alltech, now Lalemand), GERT STRAND® (available from Gert Strand AB, Sweden) and FERMOL® (available from DSM Specialties).

5

### **DISTILLATION**

As previously described, after fermentation, the resulting fluids can be distilled using, for example, a "beer column" to separate ethanol and other alcohols from the majority of water and residual solids. The vapor exiting the beer column can be, *e.g.*,  
10 35% by weight ethanol and can be fed to a rectification column. A mixture of nearly azeotropic (92.5%) ethanol and water from the rectification column can be purified to pure (99.5%) ethanol using vapor-phase molecular sieves. The beer column bottoms can be sent to the first effect of a three-effect evaporator. The rectification column reflux condenser can provide heat for this first effect. After the first effect, solids can be  
15 separated using a centrifuge and dried in a rotary dryer. A portion (25%) of the centrifuge effluent can be recycled to fermentation and the rest sent to the second and third evaporator effects. Most of the evaporator condensate can be returned to the process as fairly clean condensate with a small portion split off to waste water treatment to prevent build-up of low-boiling compounds.

20

### **HYDROCARBON-CONTAINING MATERIALS**

In other embodiments utilizing the methods and systems described herein, hydrocarbon-containing materials, for example mixed with biomass can be processed.  
25 Any process described herein can be used to treat any hydrocarbon-containing material herein described. "Hydrocarbon-containing materials," as used herein, is meant to include oil sands, oil shale, tar sands, coal dust, coal slurry, bitumen, various types of coal, and other naturally-occurring and synthetic materials that include both hydrocarbon components and solid matter. The solid matter can include rock, sand, clay, stone, silt,  
30 drilling slurry, or other solid organic and/or inorganic matter. The term can also include waste products such as drilling waste and by-products, refining waste and by-products,

or other waste products containing hydrocarbon components, such as asphalt shingling and covering, asphalt pavement, etc.

### CONVEYING SYSTEMS

5

Various conveying systems can be used to convey the biomass material, for example, as discussed, to a vault, and under an electron beam in a vault. Exemplary conveyors are belt conveyors, pneumatic conveyors, screw conveyors, carts, trains, trains or carts on rails, elevators, front loaders, backhoes, cranes, various scrapers and shovels, trucks, and throwing devices can be used. For example, vibratory conveyors can be used in various processes described herein. Vibratory conveyors are described in PCT/US2013/64289 filed October 10, 2013 the full disclosure of which is incorporated by reference herein.

Vibratory conveyors are particularly useful for spreading the material and producing a uniform layer on the conveyor trough surface. For example the initial feedstock can form a pile of material that can be at least four feet high (e.g., at least about 3 feet, at least about 2 feet, at least about 1 foot, at least about 6 inches, at least about 5 inches, at least about, 4 inches, at least about 3 inches, at least about 2 inches, at least about 1 inch, at least about ½ inch) and spans less than the width of the conveyor (e.g., less than about 10%, less than about 20%, less than about 30%, less than about 40%, less than about 50%, less than about 60%, less than about 70%, less than about 80%, less than about 90%, less than about 95%, less than about 99%). The vibratory conveyor can spread the material to span the entire width of the conveyor trough and have a uniform thickness, preferably as discussed above. In some cases, an additional spreading method can be useful. For example, a spreader such as a broadcast spreader, a drop spreader (e.g., a CHRISTY SPREADER™) or combinations thereof can be used to drop (e.g., place, pour, spill and/or sprinkle) the feedstock over a wide area. Optionally, the spreader can deliver the biomass as a wide shower or curtain onto the vibratory conveyor. Additionally, a second conveyor, upstream from the first conveyor (e.g., the first conveyor is used in the irradiation of the feedstock), can drop biomass onto the first conveyor, where the second conveyor can have a width transverse to the direction of

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conveying smaller than the first conveyor. In particular, when the second conveyor is a vibratory conveyor, the feedstock is spread by the action of the second and first conveyor. In some optional embodiments, the second conveyor ends in a bias cross cut discharge (e.g., a bias cut with a ratio of 4:1) so that the material can be dropped as a wide curtain (e.g., wider than the width of the second conveyor) onto the first conveyor. The initial drop area of the biomass by the spreader (e.g., broadcast spreader, drop spreader, conveyor, or cross cut vibratory conveyor) can span the entire width of the first vibratory conveyor, or it can span part of this width. Once dropped onto the conveyor, the material is spread even more uniformly by the vibrations of the conveyor so that, preferably, the entire width of the conveyor is covered with a uniform layer of biomass. In some embodiments combinations of spreaders can be used. Some methods of spreading a feed stock are described in U.S. Patent No. 7,153,533, filed July 23, 2002 and published December 26, 2006, the entire disclosure of which is incorporated herein by reference.

Generally, it is preferred to convey the material as quickly as possible through an electron beam to maximize throughput. For example, the material can be conveyed at rates of at least 1 ft/min, e.g., at least 2 ft/min, at least 3 ft/min, at least 4 ft/min, at least 5 ft/min, at least 10 ft/min, at least 15 ft/min, at least 20 ft/min, at least 25 ft/min, at least 30 ft/min, at least 40 ft/min, at least 50 ft/min, at least 60 ft/min, at least 70 ft/min, at least 80 ft/min, at least 90 ft/min. The rate of conveying is related to the beam current and targeted irradiation dose, for example, for a ¼ inch thick biomass spread over a 5.5 foot wide conveyor and 100 mA, the conveyor can move at about 20 ft/min to provide a useful irradiation dosage (e.g. about 10 Mrad for a single pass), at 50 mA the conveyor can move at about 10 ft/min to provide approximately the same irradiation dosage.

The rate at which material can be conveyed depends on the shape and mass of the material being conveyed, and the desired treatment. Flowing materials e.g., particulate materials, are particularly amenable to conveying with vibratory conveyors. Conveying speeds can, for example be, at least 100 lb/hr (e.g., at least 500 lb/hr, at least 1000 lb/hr, at least 2000 lb/hr, at least 3000 lb/hr, at least 4000 lb/hr, at least 5000 lb/hr, at least 10,000 lb/hr, at least 15,000 lb/hr, or even at least 25,000 lb/hr). Some typical conveying speeds can be between about 1000 and 10,000 lb/hr, (e.g., between about 1000 lb/hr and



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8000 lb/hr, between about 2000 and 7000 lb/hr, between about 2000 and 6000 lb/hr, between about 2000 and 5000 lb/hr, between about 2000 and 4500 lb/hr, between about 1500 and 5000 lb/hr, between about 3000 and 7000 lb/hr, between about 3000 and 6000 lb/hr, between about 4000 and 6000 lb/hr and between about 4000 and 5000 lb/hr).

- 5 Typical conveying speeds depend on the density of the material. For example, for a biomass with a density of about 35 lb/ft<sup>3</sup>, and a conveying speed of about 5000 lb/hr, the material is conveyed at a rate of about 143 ft<sup>3</sup>/hr, if the material is ¼” thick and is in a trough 5.5 ft wide, the material is conveyed at a rate of about 1250 ft/hr (about 21 ft/min). Rates of conveying the material can therefore vary greatly. Preferably, for
- 10 example, a ¼” thick layer of biomass, is conveyed at speeds of between about 5 and 100 ft/min (e.g. between about 5 and 100 ft/min, between about 6 and 100 ft/min, between about 7 and 100 ft/min, between about 8 and 100 ft/min, between about 9 and 100 ft/min, between about 10 and 100 ft/min, between about 11 and 100 ft/min, between about 12 and 100 ft/min, between about 13 and 100 ft/min, between about 14 and 100 ft/min,
- 15 between about 15 and 100 ft/min, between about 20 and 100 ft/min, between about 30 and 100 ft/min, between about 40 and 100 ft/min, between about 2 and 60 ft/min, between about 3 and 60 ft/min, between about 5 and 60 ft/min, between about 6 and 60 ft/min, between about 7 and 60 ft/min, between about 8 and 60 ft/min, between about 9 and 60 ft/min, between about 10 and 60 ft/min, between about 15 and 60 ft/min, between
- 20 about 20 and 60 ft/min, between about 30 and 60 ft/min, between about 40 and 60 ft/min, between about 2 and 50 ft/min, between about 3 and 50 ft/min, between about 5 and 50 ft/min, between about 6 and 50 ft/min, between about 7 and 50 ft/min, between about 8 and 50 ft/min, between about 9 and 50 ft/min, between about 10 and 50 ft/min, between about 15 and 50 ft/min, between about 20 and 50 ft/min, between about 30 and 50 ft/min,
- 25 between about 40 and 50 ft/min). It is preferable that the material be conveyed at a constant rate, for example, to help maintain a constant irradiation of the material as it passes under the electron beam (e.g., shower, field).

The vibratory conveyors described can include screens used for sieving and sorting materials. Port openings on the side or bottom of the troughs can be used for

30 sorting, selecting or removing specific materials, for example, by size or shape. Some conveyors have counterbalances to reduce the dynamic forces on the support structure.

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Some vibratory conveyors are configured as spiral elevators, are designed to curve around surfaces and/or are designed to drop material from one conveyor to another (e.g., in a step, cascade or as a series of steps or a stair). Along with conveying materials conveyors can be used, by themselves or coupled with other equipment or systems, for  
5 screening, separating, sorting, classifying, distributing, sizing, inspection, picking, metal removing, freezing, blending, mixing, orienting, heating, cooking, drying, dewatering, cleaning, washing, leaching, quenching, coating, de-dusting and/or feeding. The conveyors can also include covers (e.g., dust-tight covers), side discharge gates, bottom discharge gates, special liners (e.g., anti-stick, stainless steel, rubber, custom steel, and or  
10 grooved), divided troughs, quench pools, screens, perforated plates, detectors (e.g., metal detectors), high temperature designs, food grade designs, heaters, dryers and or coolers. In addition, the trough can be of various shapes, for example, flat bottomed, vee shaped bottom, flanged at the top, curved bottom, flat with ridges in any direction, tubular, half pipe, covered or any combinations of these. In particular, the conveyors can be coupled  
15 with an irradiation systems and/or equipment.

The conveyors (e.g., vibratory conveyor) can be made of corrosion resistant materials. The conveyors can utilize structural materials that include stainless steel (e.g., 304, 316 stainless steel, HASTELLOY® ALLOYS and INCONEL® Alloys). For example, HASTELLOY® Corrosion-Resistant alloys from Hynes (Kokomo, Indiana,  
20 USA) such as HASTELLOY® B-3® ALLOY, HASTELLOY® HYBRID-BC1® ALLOY, HASTELLOY® C-4 ALLOY, HASTELLOY® C-22® ALLOY, HASTELLOY® C-22HS® ALLOY, HASTELLOY® C-276 ALLOY, HASTELLOY® C-2000® ALLOY, HASTELLOY® G-30® ALLOY, HASTELLOY® G-35® ALLOY, HASTELLOY® N ALLOY and HASTELLOY® ULTIMET® alloy.

25 The vibratory conveyors can include non-stick release coatings, for example, TUFFLON™ (Dupont, Delaware, USA). The vibratory conveyors can also include corrosion resistant coatings. For example, coatings that can be supplied from Metal Coatings Corp (Houston, Texas, USA) and others such as Fluoropolymer, XYLAN®, Molybdenum Disulfide, Epoxy Phenolic, Phosphate- ferrous metal coating,  
30 Polyurethane- high gloss topcoat for epoxy, inorganic zinc, Poly Tetrafluoro ethylene, PPS/Ryton®, fluorinated ethylene propylene, PVDF/DYKOR®, ECTFE/HALAR®

and Ceramic Epoxy Coating. The coatings can improve resistance to process gases (e.g., ozone), chemical corrosion, pitting corrosion, galling corrosion and oxidation.

Optionally, in addition to the conveying systems described herein, one or more other conveying systems can be enclosed. When using an enclosure, the enclosed conveyor can also be purged with an inert gas so as to maintain an atmosphere at a reduced oxygen level. Keeping oxygen levels low avoids the formation of ozone which in some instances is undesirable due to its reactive and toxic nature. For example, the oxygen can be less than about 20% (e.g., less than about 10%, less than about 1%, less than about 0.1%, less than about 0.01%, or even less than about 0.001% oxygen).

Purging can be done with an inert gas including, but not limited to, nitrogen, argon, helium or carbon dioxide. This can be supplied, for example, from a boil off of a liquid source (e.g., liquid nitrogen or helium), generated or separated from air *in situ*, or supplied from tanks. The inert gas can be recirculated and any residual oxygen can be removed using a catalyst, such as a copper catalyst bed. Alternatively, combinations of purging, recirculating and oxygen removal can be done to keep the oxygen levels low.

The enclosed conveyor can also be purged with a reactive gas that can react with the biomass. This can be done before, during or after the irradiation process. The reactive gas can be, but is not limited to, nitrous oxide, ammonia, oxygen, ozone, hydrocarbons, aromatic compounds, amides, peroxides, azides, halides, oxyhalides, phosphides, phosphines, arsines, sulfides, thiols, boranes and/or hydrides. The reactive gas can be activated in the enclosure, e.g., by irradiation (e.g., electron beam, UV irradiation, microwave irradiation, heating, IR radiation), so that it reacts with the biomass. The biomass itself can be activated, for example by irradiation. Preferably the biomass is activated by the electron beam, to produce radicals which then react with the activated or unactivated reactive gas, e.g., by radical coupling or quenching.

Purging gases supplied to an enclosed conveyor can also be cooled, for example below about 25°C, below about 0°C, below about -40°C, below about -80°C, below about -120°C. For example, the gas can be boiled off from a compressed gas such as liquid nitrogen or sublimed from solid carbon dioxide. As an alternative example, the gas can be cooled by a chiller or part of or the entire conveyor can be cooled.

### OTHER EMBODIMENTS

Any material, processes or processed materials discussed herein can be used to  
5 make products and/or intermediates such as composites, fillers, binders, plastic additives,  
adsorbents and controlled release agents. The methods can include densification, for  
example, by applying pressure and heat to the materials. For example composites can be  
made by combining fibrous materials with a resin or polymer. For example radiation  
cross-linkable resin, e.g., a thermoplastic resin can be combined with a fibrous material  
10 to provide a fibrous material/cross-linkable resin combination. Such materials can be, for  
example, useful as building materials, protective sheets, containers and other structural  
materials (e.g., molded and/or extruded products). Adsorbents can be, for example, in the  
form of pellets, chips, fibers and/or sheets. Adsorbents can be used, for example, as pet  
bedding, packaging material or in pollution control systems. Controlled release matrices  
15 can also be the form of, for example, pellets, chips, fibers and or sheets. The controlled  
release matrices can, for example, be used to release drugs, biocides, fragrances. For  
example, composites, adsorbents and control release agents and their uses are described  
in International Application No. PCT/US2006/010648, filed March 23, 2006, and US  
Patent No. 8,074,910 filed November 22, 2011, the entire disclosures of which are herein  
20 incorporated by reference.

In some instances the biomass material is treated at a first level to reduce  
recalcitrance, e.g., utilizing accelerated electrons, to selectively release one or more  
sugars (e.g., xylose). The biomass can then be treated to a second level to release one or  
more other sugars (e.g., glucose). Optionally the biomass can be dried between  
25 treatments. The treatments can include applying chemical and biochemical treatments to  
release the sugars. For example, a biomass material can be treated to a level of less than  
about 20 Mrad (e.g., less than about 15 Mrad, less than about 10 Mrad, less than about 5  
Mrad, less than about 2 Mrad) and then treated with a solution of sulfuric acid,  
containing less than 10% sulfuric acid (e.g., less than about 9%, less than about 8%, less  
30 than about 7%, less than about 6%, less than about 5%, less than about 4%, less than  
about 3%, less than about 2%, less than about 1%, less than about 0.75%, less than about

0.50 %, less than about 0.25%) to release xylose. Xylose, for example that is released into solution, can be separated from solids and optionally the solids washed with a solvent/solution (e.g., with water and/or acidified water). Optionally, the solids can be dried, for example in air and/or under vacuum optionally with heating (e.g., below about  
5 150 deg C, below about 120 deg C) to a water content below about 25 wt% (below about 20 wt.%, below about 15 wt.%, below about 10 wt.%, below about 5 wt.%). The solids can then be treated with a level of less than about 30 Mrad (e.g., less than about 25 Mrad, less than about 20 Mrad, less than about 15 Mrad, less than about 10 Mrad, less than about 5 Mrad, less than about 1 Mrad or even not at all) and then treated with an enzyme  
10 (e.g., a cellulase) to release glucose. The glucose (e.g., glucose in solution) can be separated from the remaining solids. The solids can then be further processed, for example utilized to make energy or other products (e.g., lignin derived products).

An optional embodiment for filtering materials described herein includes utilizing a Rotary Pressure Filter and/or a Vacuum Belt Filter. The saccharified material is filtered  
15 using this equipment where, in general, the product sugars are separated from the solids. The equipment provides the means to wash retained sugars from the filter solids. The filter cake can be used for energy, utilized for valuable components such as lignin derived products, or recycled for further bioprocessing.

FIG. 4A shows a perspective exploded view of a Rotary Pressure Filter **400**.  
20 Filter drum **410** rotates continuously in a pressure-sealed housing **420** at infinitely variable speed, for example, in the direction indicated by the curved dashed arrows. The drum is covered with a filter cloth, for example, as previously described for rotary vacuum drum filtration apparatus. Feedstock (e.g., saccharified biomass) enters the interior of the drum through ports (shown in FIGS 4B-4E) disposed on the bottom of  
25 drum housing. The annular space between drum and housing is sealed to the sides by stuffing boxes and divided into pressure-tight segment chambers by separating elements. The surface of the drum consists of filter cells **430** in fluid connection to drainage pipes **440** through control head **450**. The feedstock is forced through the filter cells, to the drainage pipes and exits as filtered liquid product at port **414** as indicated by arrow **B**  
30 (e.g., the arrow shows the flow of a filtered sugar solution derived from the saccharified material). Arrows **A** indicate a drying gas (e.g., air, nitrogen, steam, including super

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heated steam) that enters through ports **412** as indicated by arrows **A** (e.g., arrows indicating the flow of the gas which dries the solids on the surface of the drum while forcing the liquids through the filter cells **430**). Filter cake is removed from the drum surface and exits through discharge **416**.

5           Details of how the processing through the pressure filter are shown in more detail by side view partial cross section FIGS 4B, 4C, 4D and 4E. FIG. 4B shows a step of inputting the feedstock and filtering using the filter device. A feedstock (e.g., saccharified material) is fed continuously through port **510** as indicated by the straight arrow **C**. The material is fed continuously and under pressure to the filter from below the drum. In the filter cells, a filter cake **520** forms on the filter elements and is carried into the flowing segment chambers by drum rotation (e.g., the rotation direction as indicated by the curved arrow). The filtrate drains off the cells through the pipe system, including pipes **440** to the control head. FIG. 4C shows a step of washing the filter cake. Optionally, e.g., depending on the application, washing, extraction or steaming can take  
15           place in one or more stages. As each segment chamber rotates past port **522** a washing fluid is forced through the cake and into the pipe system as indicated by the solid arrow **D**. For example, the washing fluid can be water, optionally with additives such as anti-foam agents, chelates, viscosity modified, surfactants and/or pH modifiers (e.g., acids, bases, buffers). The washing fluid can optionally be heated or cooled to aid in the  
20           extraction process, for example, steam can be used. The washing fluid is drained through the pipe system to the control head. Although it can be desirable to extract as much of the soluble components as possible (e.g., sugars), the dilution of the components should be considered *e.g.*, it may not be practical to dilute components excessively. FIG. 4D shows a cake drying step. As previously discussed a gas such as compressed air, nitrogen  
25           or steam can be made to flow through port **412** so as to dry the filter cake while forcing fluids into the tubing system and to the control head. FIG. 4E shows a step of discharging the cake from the drum surface. Discharging of the filter cake occurs in a discharge zone that is non-pressurized. The discharge zone can include a gas-tight hood if required. The cake is discharged by, for example, back-blowing and optionally, by a  
30           movable scraper. The filter cloth can then be washed. Filter cake **550** is removed through port **416**. A device for cleaning the cake, either continuously or on demand, can also be

included, *e.g.*, washing with a washing fluid, the fluid composition being as previously described.

Another optional embodiment for filtering materials described herein includes utilizing an Indexing Belt Filter (*e.g.*, a Vacuum Belt Filter). FIG. 5A shows a front schematic view of an Indexing Belt Filter **600**. This filtration system includes an endless filter belt **602** (*e.g.*, looped by tensioned rollers). The filter belt includes or is a filter cloth (*e.g.*, as previously described for rotary vacuum drum filtering). The feedstock can be fed continuously or stepwise to the belt. The belt moves in a stepwise movement in the direction indicated by the arrows. Vacuum trays **614** are fixed in place on a frame that provides support for components of the apparatus (*e.g.*, the rollers, the drive motors, the trays and the filter belt). The filter belt with the feedstock moves stepwise over the trays. Each time the belt stops, the filtrate is sucked downwards. Subsequently, the vacuum is shut off, releasing the filter belt which can be advanced (*e.g.*, indexed or stepped) again. A filter cake forms on top of the belt and can undergo further treatment by washing (co-current or counter-current), re-slurrying, steaming, extraction, vacuum drying and pressing. The wash filtrates (*e.g.*, sugar water) can be recovered individually from each vacuum tray and further processed. As shown and partially described, the filter system can be segmented into zones or phases wherein different processes occur. In phase **610** the feedstock is applied to the filter belt, phase **640** includes washing, phase **660** includes a drying step and phase **680** is a cake discharging step. The details of each step are described with reference to FIGS 5B, 5C, 5D and 5E.

The filter trays **614** are fixed to a frame and do not move with the belt. The trays are installed beneath the filter belt over the full length from the feed area **610** to before the cake discharge area **680**. The trays supply a support to the belt but allow liquids to pass through. For example, the surface in contact with the belt can include a support grid **619** to allow liquids to pass through. Located beneath the support grid are filtrate collecting channels **615**.

FIG. 5B shows a step and zone wherein a feedstock **612** (*e.g.*, saccharified biomass) is added to the surface of the belt filter **602**. Filtration occurs by the action of gravity and optionally vacuum applied under the belt as it is supported by each tray. Liquids flow *e.g.*, as shown by the empty arrow through grid surface **619** away from the

trays and can be collected. A filter cake **617** forms as a layer on the filter belt and is incrementally carried forward in the direction shown by the filled arrows once the initial evacuation step is complete.

FIG. 5C shows a washing step or zone. In this phase, soluble components in the filter cake that remain with the solid can be washed out. For example, sugars can be extracted by applying an aqueous solution. The solution can be water or water with additives. For example, additives as described previously such as anti-foam agents, chelates, viscosity modified, surfactants and/or pH modifiers (*e.g.*, acids, bases, buffers). The washing fluid can optionally be heated or cooled to aid in the extraction process, for example, even steam can be used. The fluid can be poured onto the filter cake as depicted by **642** or it can be sprayed on (*e.g.*, as droplets, a mist or even a vapor) as depicted by **644**. Although it can be desirable to extract as much of the soluble components as possible (*e.g.*, sugars), practical considerations may include not to diluting collected components excessively. The washings can be driven through the trays by a vacuum as in the previous phase. The washings flow in the direction indicated by the empty arrows and are collected for further processing (*e.g.*, combined with the first sugar water from the first phase, recycled and/or concentrated). In some optional embodiments the washings from the second phase are re-used for additional washings until a specific concentration of extracted components is reached (*e.g.*, sugars at least to 1 wt%, 2 wt%, 5 wt%, 10 wt%).

FIG. 5D shows a step and zone wherein drying can be done by applying the vacuum to the underside of the belt as previously described. Optionally, hot air or steaming hoods **662** can be used. For example, hot steam can be injected through applicator **664**. Optionally, a mechanical means of drying can be utilized. For example, pressing device **666** can be utilized. Optionally, the pressing device can be a thermal pressing device, *e.g.*, wherein the pressing device surface in contact with the filter cake, is heated. Liquids or fluids (*e.g.*, gases such as vapor) can be made to flow as indicated by the open arrows and collected for further processing or recycling as previously discussed.

FIG. 5E shows a cake discharge and belt cleaning section. The filter cake is discharged between the discharge rollers **682** and **684**. A scraper can be installed **686** that



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strips off any cake residue **688** still adhering to the belt at roller **684**. The filter cake **617** and residue **688** can be collected and further processed. A washing station **690** can wash and/or condition the filter belt before the belt loops back to the first zone **610**.

Rotary Pressure Filters and Indexing Belt filters can be purchased from BHS-  
5 Sonthofen Inc. (Charlotte, N.C.) and used as designed or modified. One or more of each of these can be utilized in the filtering methods described. For example, filtering aids can be utilized. For example, one or more of each of these can be used to replace any of the filtering elements described in FIGs 3A, 3B or 3C. For example RVDF **340**, centrifuge **350**, screener **360** can be replaced by a Rotary Pressure filter and/or an Indexing Belt  
10 Filter. Optionally or additionally one or more Rotary Pressure Filter and/or one or more Indexing Belt Filters can be used in addition to the filtering elements discussed. For example, an Indexing Belt Filter can be inserted between the screener **360** and the distillation **330** described by FIG. 3C, or a Rotary Pressure Filter can be inserted after the distillation **330** and before the RVDF **340** described in FIG. 3B.

15

### FLAVORS, FRAGRANCES AND COLORANTS

Any of the products and/or intermediates described herein, for example, produced by the processes, systems and/or equipment described herein, can be combined with  
20 flavors, fragrances, colorants and/or mixtures of these. For example, any one or more of (optionally along with flavors, fragrances and/or colorants) sugars, organic acids, fuels, polyols, such as sugar alcohols, biomass, fibers and composites can be combined with (e.g., formulated, mixed or reacted) or used to make other products. For example, one or more such product can be used to make soaps, detergents, candies, drinks (e.g., cola,  
25 wine, beer, liquors such as gin or vodka, sports drinks, coffees, teas), syrups, pharmaceuticals, adhesives, sheets (e.g., woven, none woven, filters, tissues) and/or composites (e.g., boards). For example, one or more such product can be combined with herbs, flowers, petals, spices, vitamins, potpourri, or candles. For example, the formulated, mixed or reacted combinations can have flavors/fragrances of grapefruit,  
30 orange, apple, raspberry, banana, lettuce, celery, cinnamon, chocolate, vanilla, peppermint, mint, onion, garlic, pepper, saffron, ginger, milk, wine, beer, tea, lean beef,

fish, clams, olive oil, coconut fat, pork fat, butter fat, beef bouillon, legume, potatoes, marmalade, ham, coffee and cheeses.

Flavors, fragrances and colorants can be added in any amount, such as between about 0.001 wt.% to about 30 wt.%, e.g., between about 0.01 to about 20, between about 5 0.05 to about 10, or between about 0.1 wt.% to about 5 wt.%. These can be formulated, mixed and or reacted (e.g., with any one of more product or intermediate described herein) by any means and in any order or sequence (e.g., agitated, mixed, emulsified, gelled, infused, heated, sonicated, and/or suspended). Fillers, binders, emulsifier, antioxidants can also be utilized, for example protein gels, starches and silica.

10 In one embodiment the flavors, fragrances and colorants can be added to the biomass immediately after the biomass is irradiated such that the reactive sites created by the irradiation may react with reactive compatible sites of the flavors, fragrances, and colorants.

The flavors, fragrances and colorants can be natural and/or synthetic materials. 15 These materials can be one or more of a compound, a composition or mixtures of these (e.g., a formulated or natural composition of several compounds). Optionally the flavors, fragrances, antioxidants and colorants can be derived biologically, for example, from a fermentation process (e.g., fermentation of saccharified materials as described herein). Alternatively, or additionally these flavors, fragrances and colorants can be harvested 20 from a whole organism (e.g., plant, fungus, animal, bacteria or yeast) or a part of an organism. The organism can be collected and or extracted to provide color, flavors, fragrances and/or antioxidant by any means including utilizing the methods, systems and equipment described herein, hot water extraction, supercritical fluid extraction, chemical extraction (e.g., solvent or reactive extraction including acids and bases), mechanical 25 extraction (e.g., pressing, comminuting, filtering), utilizing an enzyme, utilizing a bacteria such as to break down a starting material, and combinations of these methods. The compounds can be derived by a chemical reaction, for example, the combination of a sugar (e.g., as produced as described herein) with an amino acid (Maillard reaction). The flavor, fragrance, antioxidant and/or colorant can be an intermediate and or product 30 produced by the methods, equipment or systems described herein, for example and ester and a lignin derived product.

Some examples of flavor, fragrances or colorants are polyphenols. Polyphenols are pigments responsible for the red, purple and blue colorants of many fruits, vegetables, cereal grains, and flowers. Polyphenols also can have antioxidant properties and often have a bitter taste. The antioxidant properties make these important

5 preservatives. One class of polyphenols are the flavonoids, such as Anthocyanidines, flavanones, flavan-3-ols, s, flavanones and flavanones. Other phenolic compounds that can be used include phenolic acids and their esters, such as chlorogenic acid and polymeric tannins.

Among the colorants inorganic compounds, minerals or organic compounds can

10 be used, for example titanium dioxide, zinc oxide, aluminum oxide, cadmium yellow (E.g., CdS), cadmium orange (e.g., CdS with some Se), alizarin crimson (e.g., synthetic or non-synthetic rose madder), ultramarine (e.g., synthetic ultramarine, natural ultramarine, synthetic ultramarine violet ), cobalt blue, cobalt yellow, cobalt green, viridian (e.g., hydrated chromium(III)oxide), chalcophylite, conicalcite, cornubite,

15 cornwallite and lironite. Black pigments such as carbon black and self-dispersed blacks may be used.

Some flavors and fragrances that can be utilized include ACALEA TBHQ, ACET C-6, ALLYL AMYL GLYCOLATE, ALPHA TERPINEOL, AMBRETOLIDE, AMBRINOL 95, ANDRANE, APHERMATE, APPLELIDE, BACDANOL®,

20 BERGAMAL, BETA IONONE EPOXIDE, BETA NAPHTHYL ISO-BUTYL ETHER, BICYCLONONALACTONE, BORNAFIX®, CANTHOXAL, CASHMERAN®, CASHMERAN® VELVET, CASSIFFIX®, CEDRAFIX, CEDRAMBER®, CEDRYL ACETATE, CELESTOLIDE, CINNAMALVA, CITRAL DIMETHYL ACETATE, CITROLATE™, CITRONELLOL 700, CITRONELLOL 950, CITRONELLOL

25 COEUR, CITRONELLYL ACETATE, CITRONELLYL ACETATE PURE, CITRONELLYL FORMATE, CLARYCET, CLONAL, CONIFERAN, CONIFERAN PURE, CORTEX ALDEHYDE 50% PEOMOSA, CYCLABUTE, CYCLACET®, CYCLAPROP®, CYCLEMAX™, CYCLOHEXYL ETHYL ACETATE, DAMASCOL, DELTA DAMASCONE, DIHYDRO CYCLACET, DIHYDRO MYRCENOL,

30 DIHYDRO TERPINEOL, DIHYDRO TERPINYL ACETATE, DIMETHYL CYCLORMOL, DIMETHYL OCTANOL PQ, DIMYRCETOL, DIOLA, DIPENTENE,

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DULCINYL® RECRYSTALLIZED, ETHYL-3-PHENYL GLYCIDATE,  
 FLEURAMONE, FLEURANIL, FLORAL SUPER, FLORALOZONE, FLORIFFOL,  
 FRAISTONE, FRUCTONE, GALAXOLIDE® 50, GALAXOLIDE® 50 BB,  
 GALAXOLIDE® 50 IPM, GALAXOLIDE® UNDILUTED, GALBASCONA,  
 5 GERALDEHYDE, GERANIOL 5020, GERANIOL 600 TYPE, GERANIOL 950,  
 GERANIOL 980 (PURE), GERANIOL CFT COEUR, GERANIOL COEUR,  
 GERANYL ACETATE COEUR, GERANYL ACETATE, PURE, GERANYL  
 FORMATE, GRISALVA, GUAIYL ACETATE, HELIONAL™, HERBAC,  
 HERBALIME™, HEXADECANOLIDE, HEXALON, HEXENYL SALICYLATE CIS  
 10 3-, HYACINTH BODY, HYACINTH BODY NO. 3, HYDRATROPIC  
 ALDEHYDE.DMA, HYDROXYOL, INDOLAROME, INTRELEVEN ALDEHYDE,  
 INTRELEVEN ALDEHYDE SPECIAL, IONONE ALPHA, IONONE BETA, ISO  
 CYCLO CITRAL, ISO CYCLO GERANIOL, ISO E SUPER®, ISOBUTYL  
 QUINOLINE, JASMAL., JESSEMAL®, KHARISMAL®, KHARISMAL® SUPER,  
 15 KHUSINIL, KOAVONE®, KOHINOOL®, LIFFAROME™, LIMOXAL,  
 LINDENOL™, LYRAL®, LYRAME SUPER, MANDARIN ALD 10% TRI ETH,  
 CITR, MARITIMA, MCK CHINESE, MEIJIFF™, MELAFLEUR, MELOZONE,  
 METHYL ANTHRANILATE, METHYL IONONE ALPHA EXTRA, METHYL  
 IONONE GAMMA A, METHYL IONONE GAMMA COEUR, METHYL IONONE  
 20 GAMMA PURE, METHYL LAVENDER KETONE, MONTAVERDI®, MUGUESIA,  
 MUGUET ALDEHYDE 50, MUSK Z4, MYRAC ALDEHYDE, MYRCENYL  
 ACETATE, NECTARATE™, NEROL 900, NERYL ACETATE, OCIMENE,  
 OCTACETAL, ORANGE FLOWER ETHER, ORIVONE., ORRINIFF 25%,  
 OXASPIRANE, OZOFLEUR, PAMPLEFLEUR®, PEOMOSA, PHENOXANOL®,  
 25 PICONIA, PRECYCLEMONE B, PRENYL ACETATE, PRISMANTOL, RESEDA  
 BODY, ROSALVA, ROSAMUSK, SANJINOL, SANTALIFF™, SYVERTAL,  
 TERPINEOL, TERPINOLENE 20, TERPINOLENE 90 PQ, TERPINOLENE RECT.,  
 TERPINYL ACETATE, TERPINYL ACETATE JAX, TETRAHYDRO, MUGUOL®,  
 TETRAHYDRO MYRCENOL, TETRAMERAN, TIMBERSILK™, TOBACAROL,  
 30 TRIMOFIX® O TT, TRIPLAL®, TRISAMBER®, VANORIS, VERDOX™,  
 VERDOX™ HC, VERTENEX®, VERTENEX® HC, VERTOPIX® COEUR,

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VERTOLIFF, VERTOLIFF ISO, VIOLIFF, VIVALDIE, ZENOLIDE, ABS INDIA 75  
 PCT MIGLYOL, ABS MOROCCO 50 PCT DPG, ABS MOROCCO 50 PCT TEC,  
 ABSOLUTE FRENCH, ABSOLUTE INDIA, ABSOLUTE MD 50 PCT BB,  
 ABSOLUTE MOROCCO, CONCENTRATE PG, TINCTURE 20 PCT, AMBERGRIS,  
 5 AMBRETTE ABSOLUTE, AMBRETTE SEED OIL, ARMOISE OIL 70 PCT  
 THUYONE, BASIL ABSOLUTE GRAND VERT, BASIL GRAND VERT ABS MD,  
 BASIL OIL GRAND VERT, BASIL OIL VERVEINA, BASIL OIL VIETNAM, BAY  
 OIL TERPENELESS, BEESWAX ABS N G, BEESWAX ABSOLUTE, BENZOIN  
 RESINOID SIAM, BENZOIN RESINOID SIAM 50 PCT DPG, BENZOIN RESINOID  
 10 SIAM 50 PCT PG, BENZOIN RESINOID SIAM 70.5 PCT TEC, BLACKCURRANT  
 BUD ABS 65 PCT PG, BLACKCURRANT BUD ABS MD 37 PCT TEC,  
 BLACKCURRANT BUD ABS MIGLYOL, BLACKCURRANT BUD ABSOLUTE  
 BURGUNDY, BOIS DE ROSE OIL, BRAN ABSOLUTE, BRAN RESINOID,  
 BROOM ABSOLUTE ITALY, CARDAMOM GUATEMALA CO2 EXTRACT,  
 15 CARDAMOM OIL GUATEMALA, CARDAMOM OIL INDIA, CARROT HEART,  
 CASSIE ABSOLUTE EGYPT, CASSIE ABSOLUTE MD 50 PCT IPM, CASTOREUM  
 ABS 90 PCT TEC, CASTOREUM ABS C 50 PCT MIGLYOL, CASTOREUM  
 ABSOLUTE, CASTOREUM RESINOID, CASTOREUM RESINOID 50 PCT DPG,  
 CEDROL CEDRENE, CEDRUS ATLANTICA OIL REDIST, CHAMOMILE OIL  
 20 ROMAN, CHAMOMILE OIL WILD, CHAMOMILE OIL WILD LOW LIMONENE,  
 CINNAMON BARK OIL CEYLAN, CISTE ABSOLUTE, CISTE ABSOLUTE  
 COLORLESS, CITRONELLA OIL ASIA IRON FREE, CIVET ABS 75 PCT PG,  
 CIVET ABSOLUTE, CIVET TINCTURE 10 PCT, CLARY SAGE ABS FRENCH  
 DECOL, CLARY SAGE ABSOLUTE FRENCH, CLARY SAGE C'LESS 50 PCT PG,  
 25 CLARY SAGE OIL FRENCH, COPAIBA BALSAM, COPAIBA BALSAM OIL,  
 CORIANDER SEED OIL, CYPRESS OIL, CYPRESS OIL ORGANIC, DAVANA OIL,  
 GALBANOL, GALBANUM ABSOLUTE COLORLESS, GALBANUM OIL,  
 GALBANUM RESINOID, GALBANUM RESINOID 50 PCT DPG, GALBANUM  
 RESINOID HERCOLYN BHT, GALBANUM RESINOID TEC BHT, GENTIANE  
 30 ABSOLUTE MD 20 PCT BB, GENTIANE CONCRETE, GERANIUM ABS EGYPT  
 MD, GERANIUM ABSOLUTE EGYPT, GERANIUM OIL CHINA, GERANIUM OIL

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EGYPT, GINGER OIL 624, GINGER OIL RECTIFIED SOLUBLE, GUAIAACWOOD  
 HEART, HAY ABS MD 50 PCT BB, HAY ABSOLUTE, HAY ABSOLUTE MD 50  
 PCT TEC, HEALINGWOOD, HYSSOP OIL ORGANIC, IMMORTELLE ABS YUGO  
 MD 50 PCT TEC, IMMORTELLE ABSOLUTE SPAIN, IMMORTELLE ABSOLUTE  
 5 YUGO, JASMIN ABS INDIA MD, JASMIN ABSOLUTE EGYPT, JASMIN  
 ABSOLUTE INDIA, ASMIN ABSOLUTE MOROCCO, JASMIN ABSOLUTE  
 SAMBAC, JONQUILLE ABS MD 20 PCT BB, JONQUILLE ABSOLUTE France,  
 JUNIPER BERRY OIL FLG, JUNIPER BERRY OIL RECTIFIED SOLUBLE,  
 LABDANUM RESINOID 50 PCT TEC, LABDANUM RESINOID BB, LABDANUM  
 10 RESINOID MD, LABDANUM RESINOID MD 50 PCT BB, LAVANDIN ABSOLUTE  
 H, LAVANDIN ABSOLUTE MD, LAVANDIN OIL ABRIAL ORGANIC,  
 LAVANDIN OIL GROSSO ORGANIC, LAVANDIN OIL SUPER, LAVENDER  
 ABSOLUTE H, LAVENDER ABSOLUTE MD, LAVENDER OIL COUMARIN  
 FREE, LAVENDER OIL COUMARIN FREE ORGANIC, LAVENDER OIL  
 15 MAILLETTE ORGANIC, LAVENDER OIL MT, MACE ABSOLUTE BB,  
 MAGNOLIA FLOWER OIL LOW METHYL EUGENOL, MAGNOLIA FLOWER  
 OIL, MAGNOLIA FLOWER OIL MD, MAGNOLIA LEAF OIL, MANDARIN OIL  
 MD, MANDARIN OIL MD BHT, MATE ABSOLUTE BB, MOSS TREE ABSOLUTE  
 MD TEX IFRA 43, MOSS-OAK ABS MD TEC IFRA 43, MOSS-OAK ABSOLUTE  
 20 IFRA 43, MOSS-TREE ABSOLUTE MD IPM IFRA 43, MYRRH RESINOID BB,  
 MYRRH RESINOID MD, MYRRH RESINOID TEC, MYRTLE OIL IRON FREE,  
 MYRTLE OIL TUNISIA RECTIFIED, NARCISSE ABS MD 20 PCT BB, NARCISSE  
 ABSOLUTE FRENCH, NEROLI OIL TUNISIA, NUTMEG OIL TERPENELESS,  
 OEILLET ABSOLUTE, OLIBANUM RESINOID, OLIBANUM RESINOID BB,  
 25 OLIBANUM RESINOID DPG, OLIBANUM RESINOID EXTRA 50 PCT DPG,  
 OLIBANUM RESINOID MD, OLIBANUM RESINOID MD 50 PCT DPG,  
 OLIBANUM RESINOID TEC, OPOPONAX RESINOID TEC, ORANGE BIGARADE  
 OIL MD BHT, ORANGE BIGARADE OIL MD SCFC, ORANGE FLOWER  
 ABSOLUTE TUNISIA, ORANGE FLOWER WATER ABSOLUTE TUNISIA,  
 30 ORANGE LEAF ABSOLUTE, ORANGE LEAF WATER ABSOLUTE TUNISIA,  
 ORRIS ABSOLUTE ITALY, ORRIS CONCRETE 15 PCT IRONE, ORRIS

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CONCRETE 8 PCT IRONE, ORRIS NATURAL 15 PCT IRONE 4095C, ORRIS  
 NATURAL 8 PCT IRONE 2942C, ORRIS RESINOID, OSMANTHUS ABSOLUTE,  
 OSMANTHUS ABSOLUTE MD 50 PCT BB, PATCHOULI HEART N°3,  
 PATCHOULI OIL INDONESIA, PATCHOULI OIL INDONESIA IRON FREE,  
 5 PATCHOULI OIL INDONESIA MD, PATCHOULI OIL REDIST, PENNYROYAL  
 HEART, PEPPERMINT ABSOLUTE MD, PETITGRAIN BIGARADE OIL TUNISIA,  
 PETITGRAIN CITRONNIER OIL, PETITGRAIN OIL PARAGUAY TERPENELESS,  
 PETITGRAIN OIL TERPENELESS STAB, PIMENTO BERRY OIL, PIMENTO LEAF  
 OIL, RHODINOL EX GERANIUM CHINA, ROSE ABS BULGARIAN LOW  
 10 METHYL EUGENOL, ROSE ABS MOROCCO LOW METHYL EUGENOL, ROSE  
 ABS TURKISH LOW METHYL EUGENOL, ROSE ABSOLUTE, ROSE ABSOLUTE  
 BULGARIAN, ROSE ABSOLUTE DAMASCENA, ROSE ABSOLUTE MD, ROSE  
 ABSOLUTE MOROCCO, ROSE ABSOLUTE TURKISH, ROSE OIL BULGARIAN,  
 ROSE OIL DAMASCENA LOW METHYL EUGENOL, ROSE OIL TURKISH,  
 15 ROSEMARY OIL CAMPHOR ORGANIC, ROSEMARY OIL TUNISIA,  
 SANDALWOOD OIL INDIA, SANDALWOOD OIL INDIA RECTIFIED,  
 SANTALOL, SCHINUS MOLLE OIL, ST JOHN BREAD TINCTURE 10 PCT,  
 STYRAX RESINOID, STYRAX RESINOID, TAGETE OIL, TEA TREE HEART,  
 TONKA BEAN ABS 50 PCT SOLVENTS, TONKA BEAN ABSOLUTE, TUBEROSE  
 20 ABSOLUTE INDIA, VETIVER HEART EXTRA, VETIVER OIL HAITI, VETIVER  
 OIL HAITI MD, VETIVER OIL JAVA, VETIVER OIL JAVA MD, VIOLET LEAF  
 ABSOLUTE EGYPT, VIOLET LEAF ABSOLUTE EGYPT DECOL, VIOLET LEAF  
 ABSOLUTE FRENCH, VIOLET LEAF ABSOLUTE MD 50 PCT BB, WORMWOOD  
 OIL TERPENELESS, YLANG EXTRA OIL, YLANG III OIL and combinations of  
 25 these.

The colorants can be among those listed in the Color Index International by the  
 Society of Dyers and Colourists. Colorants include dyes and pigments and include those  
 commonly used for coloring textiles, paints, inks and inkjet inks. Some colorants that  
 can be utilized include carotenoids, arylide yellows, diarylide yellows,  $\beta$ -naphthols,  
 30 naphthols, benzimidazolones, disazo condensation pigments, pyrazolones, nickel azo  
 yellow, phthalocyanines, quinacridones, perylenes and perinones, isoindolinone and

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isoindoline pigments, triarylcarbonium pigments, diketopyrrolo-pyrrole pigments, thioindigoids. Carotenoids include, for example, alpha-carotene, beta-carotene, gamma-carotene, lycopene, lutein and astaxanthin Annatto extract, Dehydrated beets (beet powder), Canthaxanthin, Caramel,  $\beta$ -Apo-8'-carotenal, Cochineal extract, Carmine,

5 Sodium copper chlorophyllin, Toasted partially defatted cooked cottonseed flour, Ferrous gluconate, Ferrous lactate, Grape color extract, Grape skin extract (enocianina), Carrot oil, Paprika, Paprika oleoresin, Mica-based pearlescent pigments, Riboflavin, Saffron, Titanium dioxide, Tomato lycopene extract; tomato lycopene concentrate,

10 Turmeric, Turmeric oleoresin, FD&C Blue No. 1, FD&C Blue No. 2, FD&C Green No. 3, Orange B, Citrus Red No. 2, FD&C Red No. 3, FD&C Red No. 40, FD&C Yellow No. 5, FD&C Yellow No. 6, Alumina (dried aluminum hydroxide), Calcium carbonate, Potassium sodium copper chlorophyllin (chlorophyllin-copper complex),

Dihydroxyacetone, Bismuth oxychloride, Ferric ammonium ferrocyanide, Ferric ferrocyanide, Chromium hydroxide green, Chromium oxide greens, Guanine,

15 Pyrophyllite, Talc, Aluminum powder, Bronze powder, Copper powder, Zinc oxide, D&C Blue No. 4, D&C Green No. 5, D&C Green No. 6, D&C Green No. 8, D&C Orange No. 4, D&C Orange No. 5, D&C Orange No. 10, D&C Orange No. 11, FD&C Red No. 4, D&C Red No. 6, D&C Red No. 7, D&C Red No. 17, D&C Red No. 21, D&C Red No. 22, D&C Red No. 27, D&C Red No. 28, D&C Red No. 30, D&C Red No. 31,

20 D&C Red No. 33, D&C Red No. 34, D&C Red No. 36, D&C Red No. 39, D&C Violet No. 2, D&C Yellow No. 7, Ext. D&C Yellow No. 7, D&C Yellow No. 8, D&C Yellow No. 10, D&C Yellow No. 11, D&C Black No. 2, D&C Black No. 3 (3), D&C Brown No. 1, Ext. D&C, Chromium-cobalt-aluminum oxide, Ferric ammonium citrate, Pyrogallol, Logwood extract, 1,4-Bis[(2-hydroxy-ethyl)amino]-9,10-anthracenedione bis(2-

25 propenoic)ester copolymers, 1,4-Bis [(2-methylphenyl)amino] -9,10-anthracenedione, 1,4-Bis[4-(2-methacryloxyethyl) phenylamino] anthraquinone copolymers, Carbazole violet, Chlorophyllin-copper complex, Chromium-cobalt-aluminum oxide,, C.I. Vat Orange 1, 2-[[2,5-Diethoxy- 4-[(4-methylphenyl)thiol] phenyl]azo] -1,3,5-benzenetriol, 16,23-Dihydrodinaphtho [2,3-a:2',3'-i] naphth [2',3':6,7] indolo [2,3-c] carbazole-

30 5,10,15,17,22,24-hexone, N,N'-(9,10-Dihydro- 9,10-dioxo- 1,5-anthracenediyl) bisbenzamide, 7,16-Dichloro- 6,15-dihydro- 5,9,14,18-anthrazinetetrone, 16,17-



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Dimethoxydinaphtho (1,2,3-cd:3',2',1'-lm) perylene-5,10-dione, Poly(hydroxyethyl methacrylate) -dye copolymers(3), Reactive Black 5, Reactive Blue 21, Reactive Orange 78, Reactive Yellow 15, Reactive Blue No. 19, Reactive Blue No. 4, C.I. Reactive Red 11, C.I. Reactive Yellow 86, C.I. Reactive Blue 163, C.I. Reactive Red 180, 4-[(2,4-  
5 dimethylphenyl)azo]- 2,4-dihydro- 5-methyl-2-phenyl- 3H-pyrazol-3-one (solvent Yellow 18), 6-Ethoxy-2- (6-ethoxy-3-oxobenzo[b] thien-2(3H)- ylidene) benzo[b]thiophen- 3(2H)-one, Phthalocyanine green, Vinyl alcohol/methyl methacrylate-dye reaction products, C.I. Reactive Red 180, C.I. Reactive Black 5, C.I. Reactive Orange 78, C.I. Reactive Yellow 15, C.I. Reactive Blue 21, Disodium 1-amino-4-[[4-[(2-  
10 bromo-1-oxoallyl)amino]-2-sulphonatophenyl]amino]-9,10-dihydro-9,10-dioxoanthracene-2-sulphonate (Reactive Blue 69), D&C Blue No. 9, [Phthalocyaninato(2-)] copper and mixtures of these.

## EXAMPLES

### Saccharification

Saccharified batches were produced as described here and used in the following  
5 examples 1 and 2.

A cylindrical tank with a diameter of 32 Inches, 64 Inches in height and fit with ASME dished heads (top and bottom) was used in the saccharification. The tank was also equipped with a hydrofoil mixing blade 16" wide. Heating was provided by flowing hot water through a half pipe jacket surrounding the tank.

10 The tank was charged with 200 Kg water, 80 Kg of biomass, and 18 Kg of DUET™ Cellulase enzyme. Biomass was corn cob that had been hammer milled and screened to a size of between 40 and 10 mesh. The biomass was irradiated with an electron beam to a total dosage of 35 Mrad. The pH of the mixture was adjusted and maintained automatically throughout the saccharification at 4.8 using Ca(OH)<sub>2</sub>. This  
15 combination was heated to 53 °C, stirred at 180 rpm (1.8 Amp at 460V) for about 24 hours after which the saccharification was considered completed

### Example 1, Centrifuge followed by Rotary Vacuum Drum Filtration

Solids were separated from saccharified batches using a continuous scroll  
20 decanter centrifuge with a 12 foot long drum. Centrifugation was performed immediately after the completion of saccharification at less than 60°C. Rates of centrifugation were 30 gallons per minute with solids loading at 15 wt.%. Solids were free of standing liquid and tested at 50 wt.% (water) on drying or less. The liquids were used for fermentation with yeast, producing ethanol. Distillation was used to separate the ethanol from the  
25 other products. The liquids sent to fermentation contained about 5 wt.% solids.

A rotary vacuum drum filter (RVDF) was used for filtering solid residues after the distillation. The 2 feet wide by 2 feet diameter drum (with approximately 6 square feet of filter cloth) was pre-coated with 2" of filter-aid. The feed stream of the distillation bottoms was 600 liters (5 wt.% solids) in 2 hours and used less than 1/10<sup>th</sup> the filter-aid  
30 from the pre-coating. The average knife advance speed was therefore less than 0.1"/hr. The filtrate contained less than 0.1wt% solids (total suspended solids, TSS). Particles

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down to about 0.5um (mean particle size) were removed. The turbidity was also very low, estimated at below about 5 NTU.

**Example 2, Vibratory screener followed by Rotary Vacuum Drum**

5 A vibratory screener with 60 mesh screens was used to remove solids from a saccharified material. This method leaves a small amount of fines in the stream. Solids were about 5 wt.% after this step. The screened material was fermented and then distilled. The distillation bottoms were filtered by RVDF under the same operating conditions as outlined above, rendering a filtered product with similar solids and  
10 turbidity relative to those given above.

Other than in the examples herein, or unless otherwise expressly specified, all of the numerical ranges, amounts, values and percentages, such as those for amounts of materials, elemental contents, times and temperatures of reaction, ratios of amounts, and  
15 others, in the following portion of the specification and attached claims may be read as if prefaced by the word “about” even though the term “about” may not expressly appear with the value, amount, or range. Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and attached claims are approximations that may vary depending upon the desired properties sought to be  
20 obtained by the present invention. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

Notwithstanding that the numerical ranges and parameters setting forth the broad  
25 scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contains error necessarily resulting from the standard deviation found in its underlying respective testing measurements. Furthermore, when numerical ranges are set forth herein, these ranges are inclusive of the recited range end points (e.g.,, end  
30 points may be used). When percentages by weight are used herein, the numerical values reported are relative to the total weight.

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Also, it should be understood that any numerical range recited herein is intended to include all sub-ranges subsumed therein. For example, a range of “1 to 10” is intended to include all sub-ranges between (and including) the recited minimum value of 1 and the recited maximum value of 10, that is, having a minimum value equal to or  
5 greater than 1 and a maximum value of equal to or less than 10. The terms “one,” “a,” or “an” as used herein are intended to include “at least one” or “one or more,” unless otherwise indicated.

Any patent, publication, or other disclosure material, in whole or in part, that is said to be incorporated by reference herein is incorporated herein only to the extent that  
10 the incorporated material does not conflict with existing definitions, statements, or other disclosure material set forth in this disclosure. As such, and to the extent necessary, the disclosure as explicitly set forth herein supersedes any conflicting material incorporated herein by reference. Any material, or portion thereof, that is said to be incorporated by  
15 reference herein, but which conflicts with existing definitions, statements, or other disclosure material set forth herein will only be incorporated to the extent that no conflict arises between that incorporated material and the existing disclosure material.

While this invention has been particularly shown and described with references to preferred embodiments thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the  
20 scope of the invention encompassed by the appended claims.

**CLAIMS**

1. A method of separating solids from liquids of a slurry, the method comprising:  
saccharifying a biomass and applying the saccharified biomass to a surface (e.g.,  
5 the outer portion) of a filter device.

2. The method of claim 1, wherein the filter device is a rotary drum filter device.

3. The method of claim 1 or 2, wherein the saccharified biomass comprises  
10 sugars selected from the group consisting of glucose, xylose and mixtures thereof.

4. The method of any one of the above claims wherein the saccharified biomass  
comprises an aqueous solvent.

15 5. The method of any one of the above claims, wherein the saccharified biomass  
comprises a non-aqueous solvent.

6. The method of any one of the above claims, wherein the saccharified biomass  
includes cells.

20

7. The method of any one of the above claims, wherein the saccharified biomass  
includes cells selected from the group consisting of yeast cells, bacterial cells, fungal  
cells or mixtures thereof.

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8. The method of any one of the above claims, wherein the saccharified biomass includes protein material.

5 9. The method of claim 8, wherein the protein material is substantially denatured protein material.

10 10. The method of claim 8 or 9, wherein the protein material includes an enzyme material.

11. The method of any one of the above claims, wherein the saccharified biomass includes a fermentation product.

12. The method of any one of the above claims, wherein the saccharified biomass includes an alcohol.

15

13. The method of claim 12, wherein the alcohol is ethanol or butanol.

14. The method of any one of claims 1 through 11, wherein the saccharified biomass includes an organic acid.

20

15. The method of claims 14, wherein the saccharified biomass includes butyric acid.

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16. The method of any one of the above claims, wherein the saccharified biomass is substantially devoid of one or more soluble sugars from which the biomass is composed.

5 17. The method of claim 16, wherein the saccharified biomass is substantially devoid of glucose.

18. The method of claims 16 or 17, wherein the saccharide biomass is substantially devoid of xylose.

10

19. The method of any one of the above claims, wherein the biomass has been saccharified using one or more saccharification agent.

15 20. The method of claim 19, wherein the saccharification agent is selected from the group consisting of enzymes, acids, bases, oxidants and mixtures thereof.

21. The method of claim 19, wherein the saccharification agent includes sulfuric acid and an enzyme.

20 22. The method of claim 19, wherein the saccharification agent is a cellulolytic enzyme.

23. The method of any one of the above claims, wherein the biomass has been treated to reduce its recalcitrance prior to saccharification.

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24. The method of claim 23, wherein recalcitrance has been reduced by the application of electron beam radiation to the biomass.

5           25. The method of any one of the above claims, wherein the filter device comprises a rotatable drum having a filter face through which liquid of the saccharified material can pass, while retaining solids of the saccharified material thereon.

10           26. The method of claim 25, wherein the filter face has a filter aid extending outwardly therefrom.

27. The method of claim 26, wherein the filter aid covers substantially the entire filter face of the rotatable drum.

15           28. The method of claim 26 or 27, wherein the filter aid is selected from the group consisting of diatomaceous earth, celite, silica, pumice, perlite, alumina, zeolites, sand, cellulosic material, lignocellulosic material, and mixtures thereof.

20           29. The method of any one of claims 26 through 28, wherein the filter aid extends from the filter face a distance of from between about 0.5 mm to about 250 mm.

30. The method of any one of claims 26 through 28, wherein the filter aid extends from the filter face a distance of from between about 1 mm and about 100 mm.



31. The method of any one of claims 26 through 28, wherein the filter aid extends from the filter face a distance of from between about 1 mm to about 50 mm.

5 32. The method of any one of claims 26 through 28, wherein the filter aid extends from the filter face a distance of from between about 2 mm to about 25 mm.

33. The method of any one of claims 25 through 33, wherein the rotatable drum includes an inner portion and wherein the inner portion is maintained at a lower pressure than the filter face.

10

34. The method of claim 33, wherein the pressure difference between the inner portion and the filter face is maintained between about 20 and about 25 inches of Hg.

15 35. The method of claim 33 or 34, wherein the inner portion is in communication with a vacuum pump and/or a vacuum source.

36. The method of any one of claims 26 through 35, wherein the filter face includes a filter cloth.

20 37. The method of claim 36, wherein the filter cloth is a woven cloth.

38. The method of claim 37, wherein the cloth has a weave selected from the group consisting of a twill weave, a plain weave, a satin weave, a knot weave, a basket weave, an oxford weave and combinations of these.

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39. The method of any one of claims 36 to 38, wherein the filter cloth has a porosity rating range from about 1 to 100 CFM/ft<sup>2</sup> can be utilized (e.g., about 1-3, about 1-10, about 10-30, about 15-20, about 30-50, about 30-40, about 50 to 70).

5

40. The method of any one of claims 26 to 39, wherein the filter device includes a knife for continuously removing the solids deposited on the drum face as the drum is rotated relative to the knife.

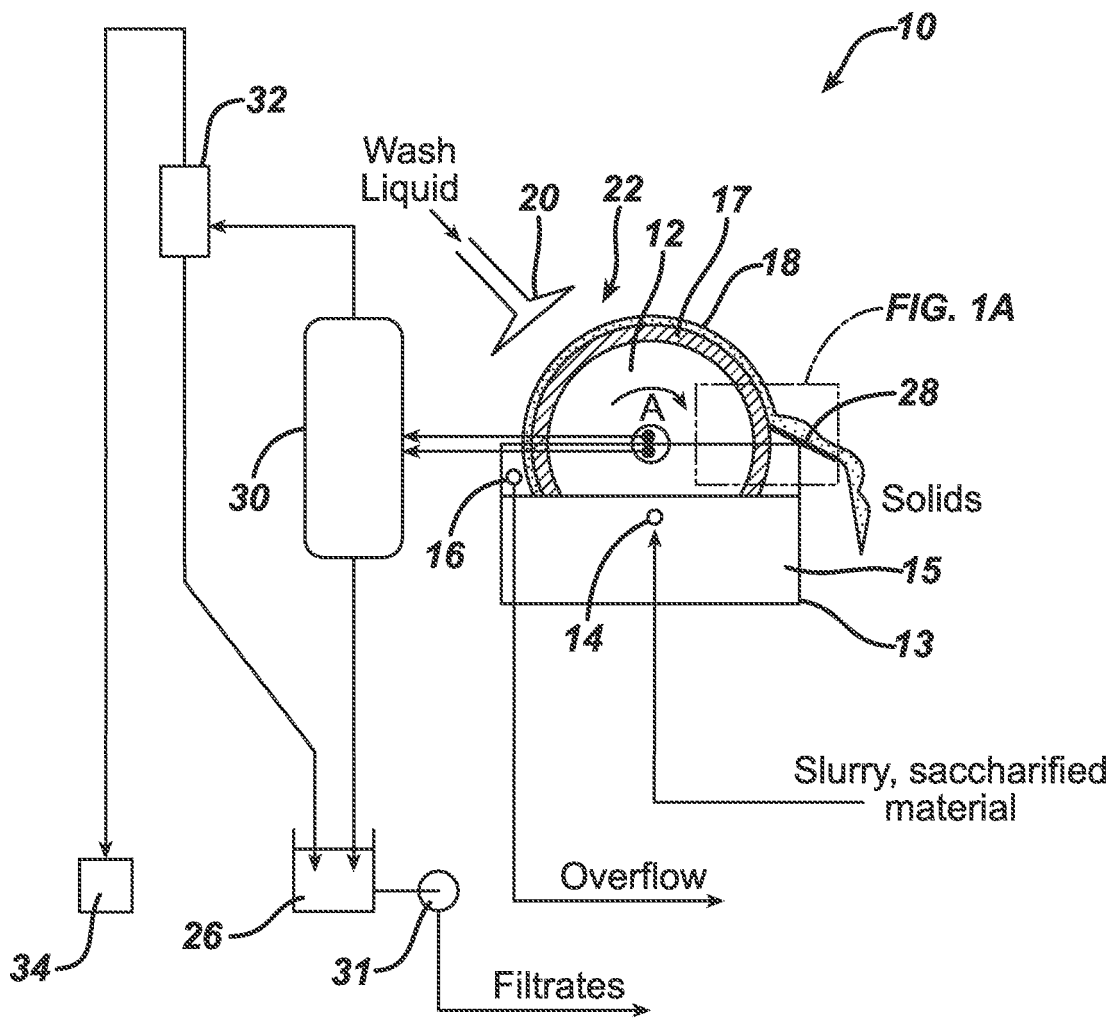
10

41. The method of claim 40, wherein the knife moves in a direction perpendicularly towards the drum face at a rate adjusted to continuously removes an interfacial region comprising the solids of the saccharified material and the filter aid.

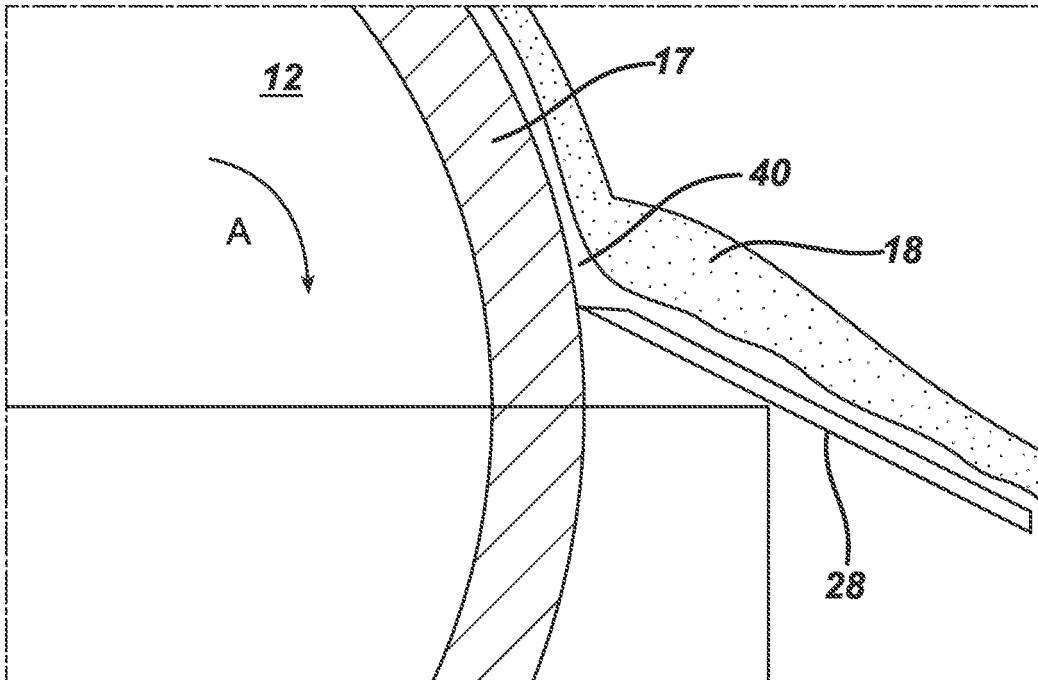
15

42. The methods of any one of the above claims, further comprising utilizing a vibratory screener to remove solids from the saccharified biomass prior to and/or after applying the saccharified biomass to the outer portion of the filter device.

FIG. 1

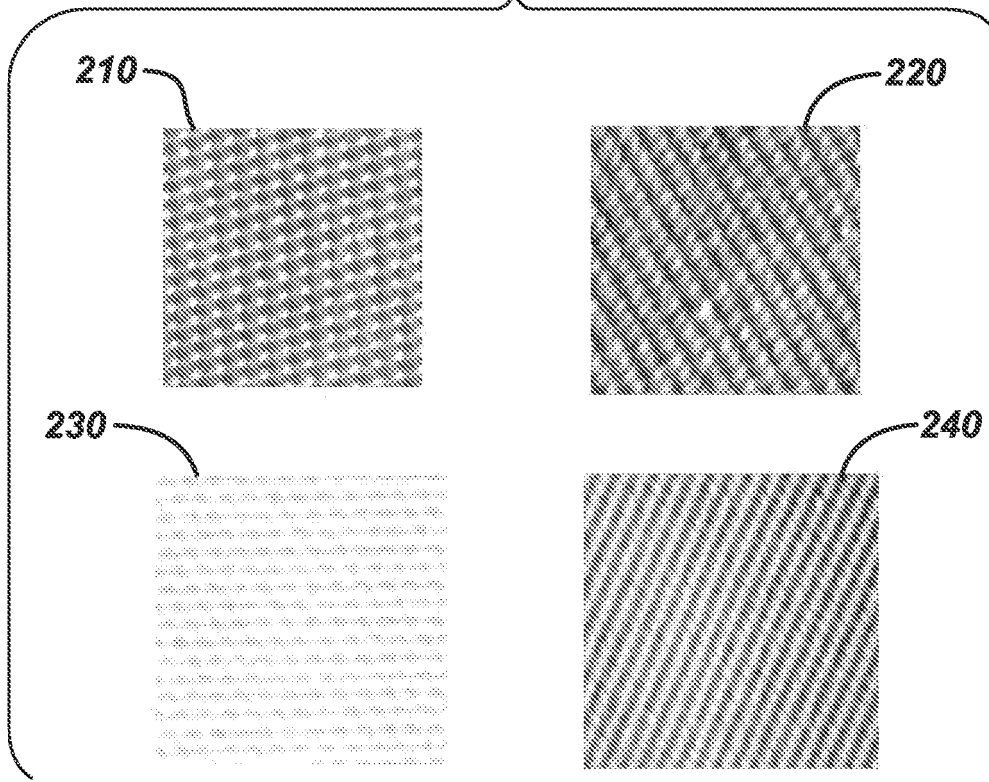


**FIG. 1A**

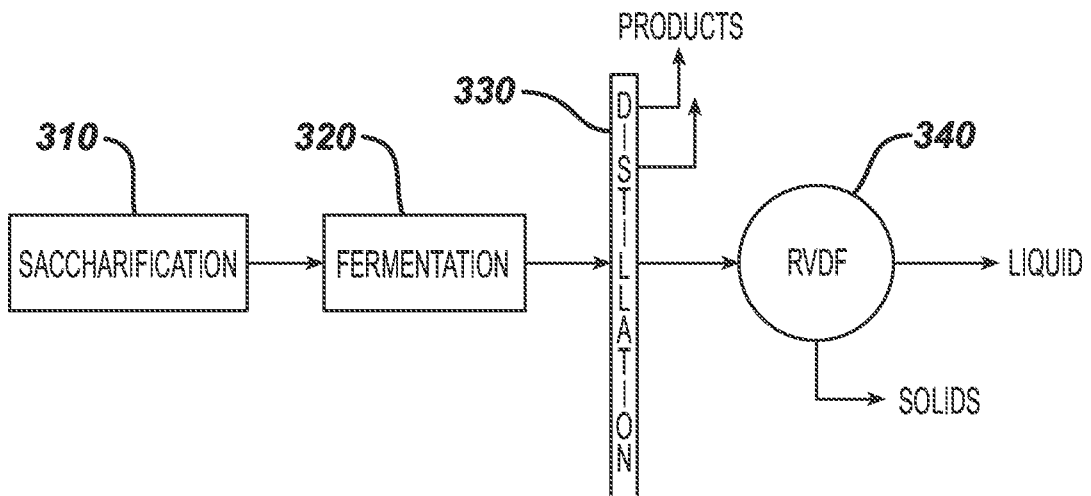


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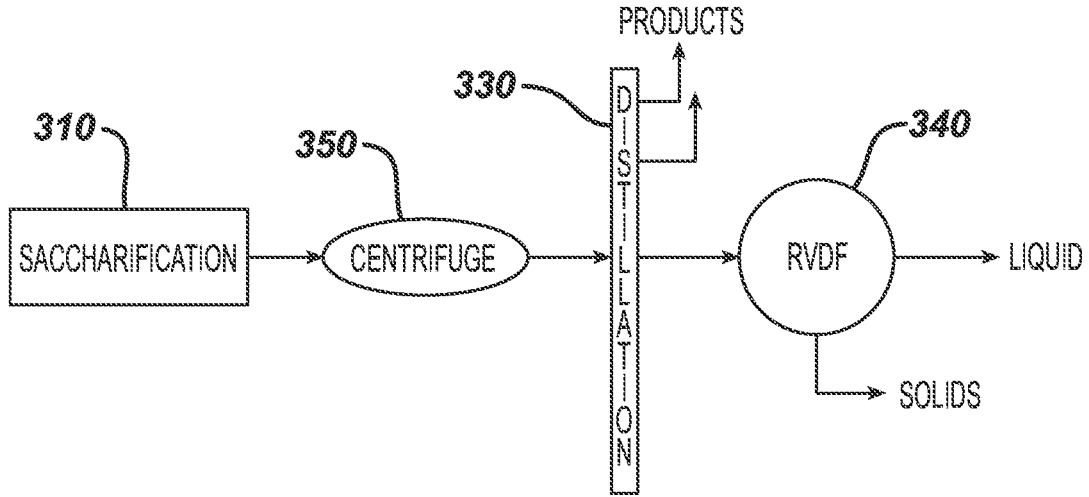
**FIG. 2**



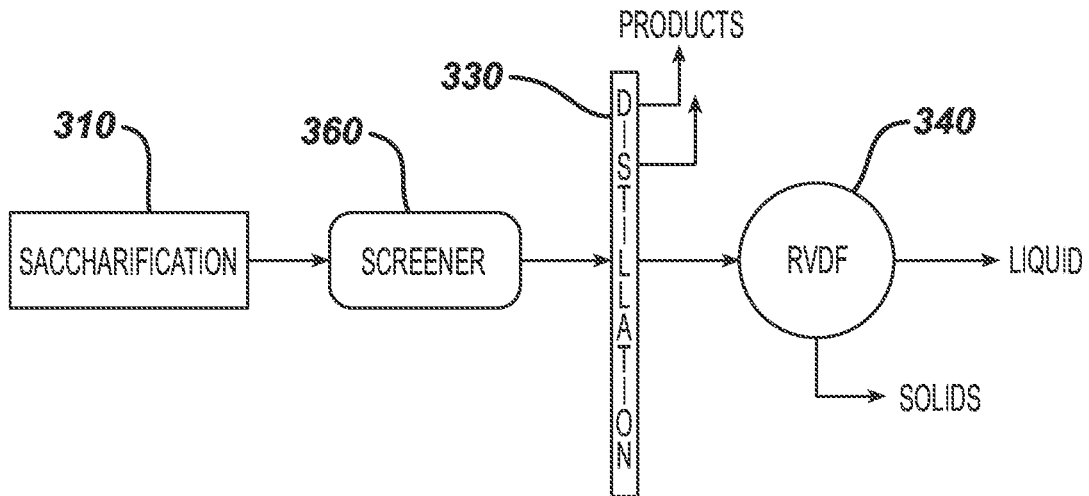
**FIG. 3A**



**FIG. 3B**

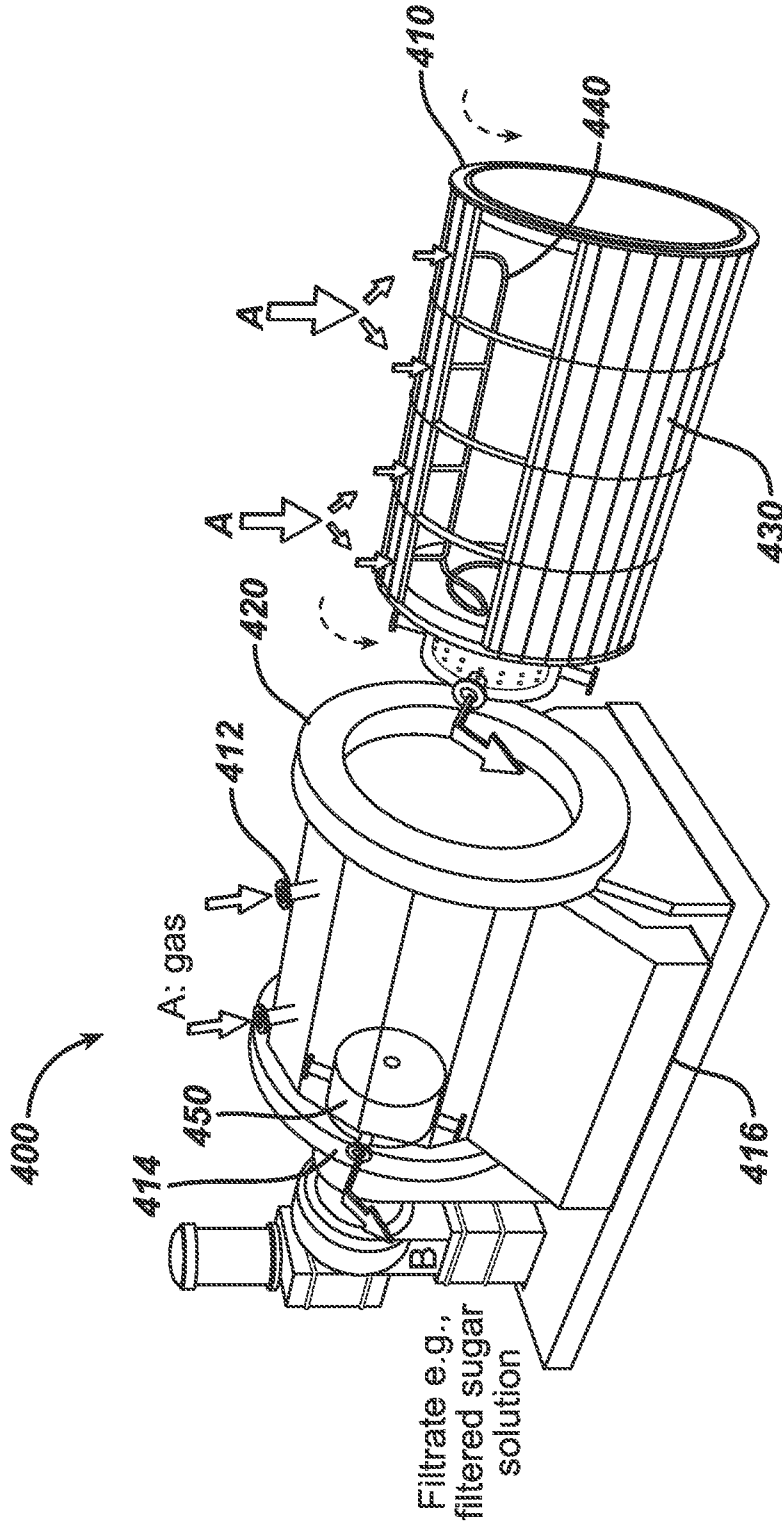


**FIG. 3C**



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FIG. 4A



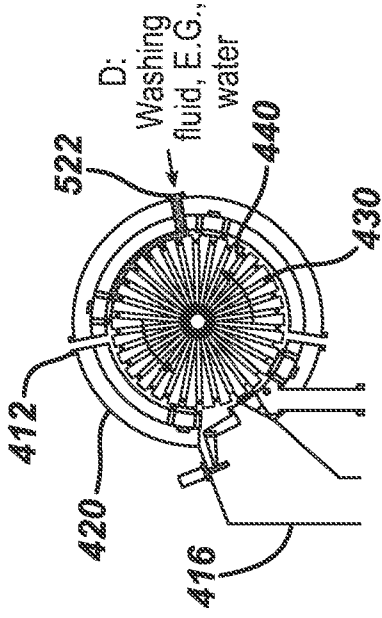


FIG. 4C

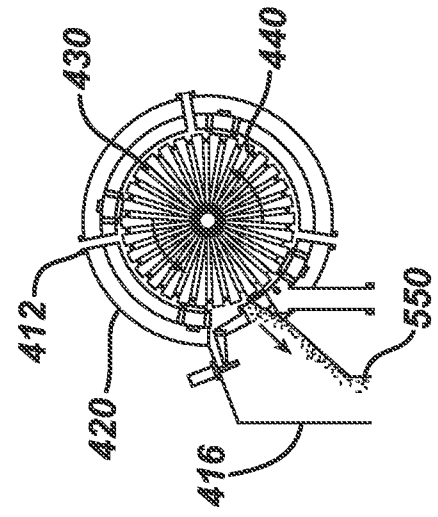


FIG. 4E

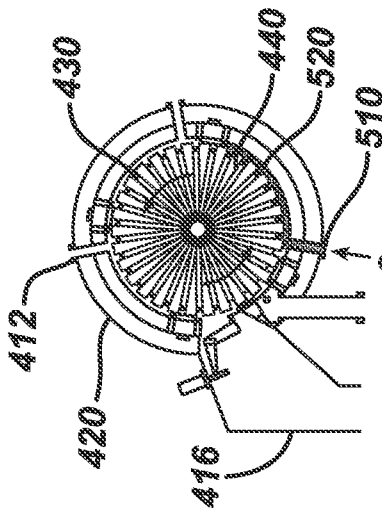


FIG. 4B

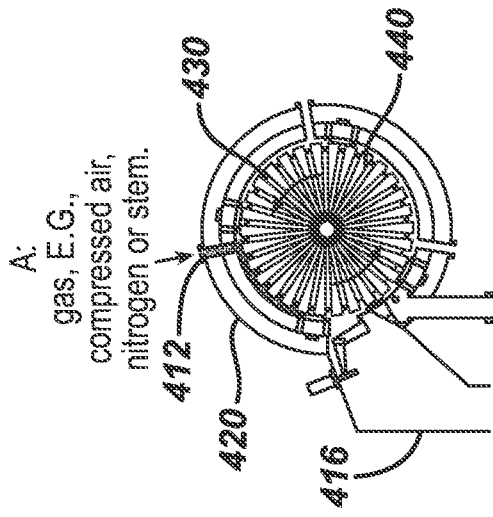
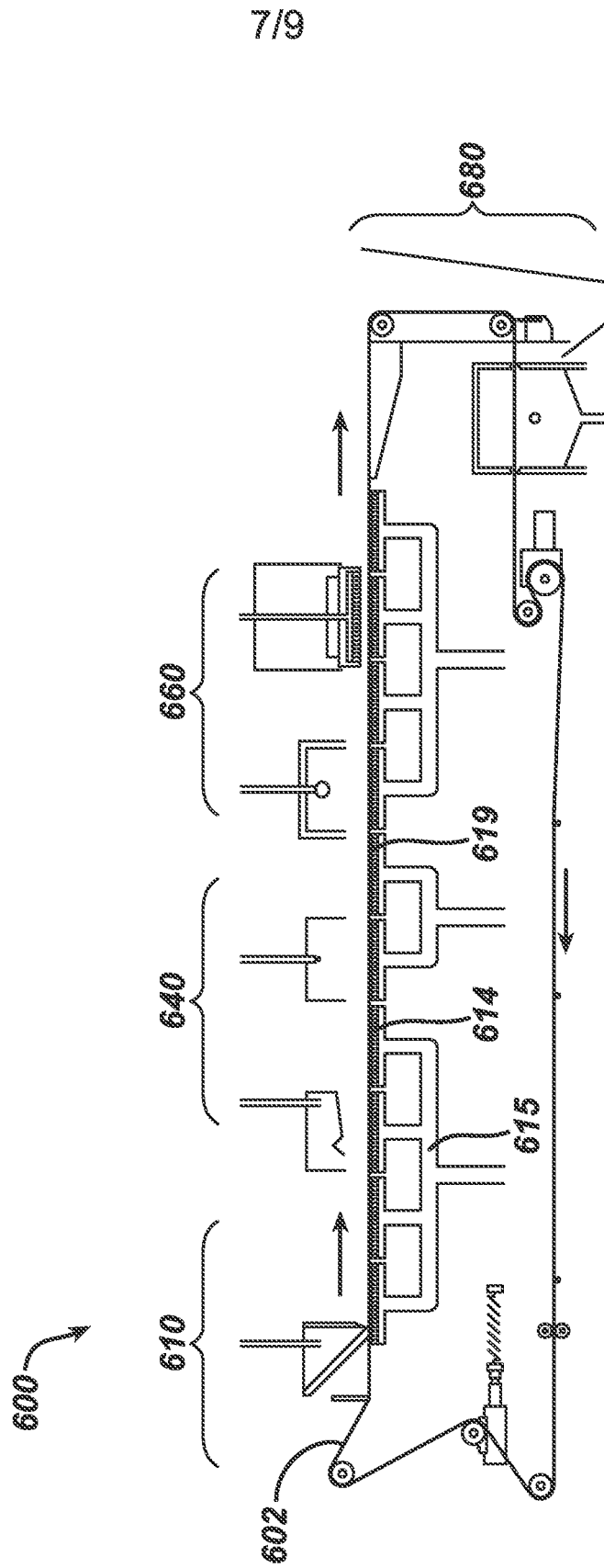


FIG. 4D

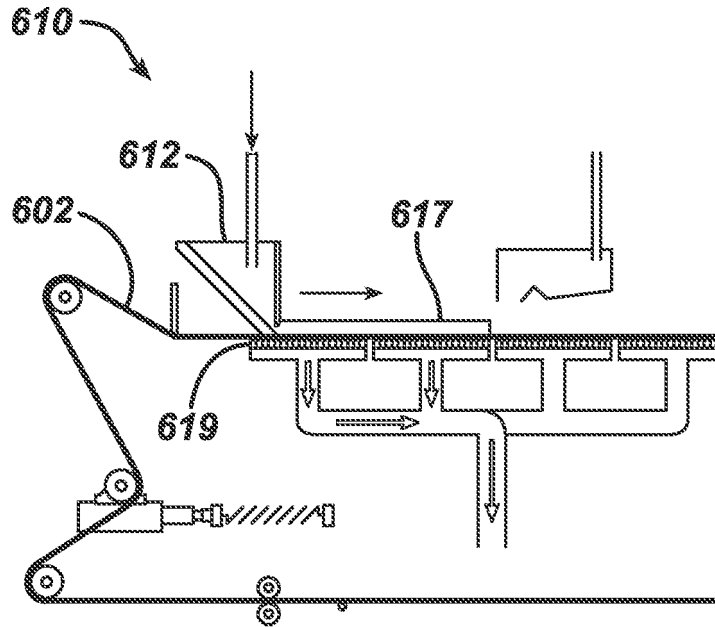


FIG. 5A

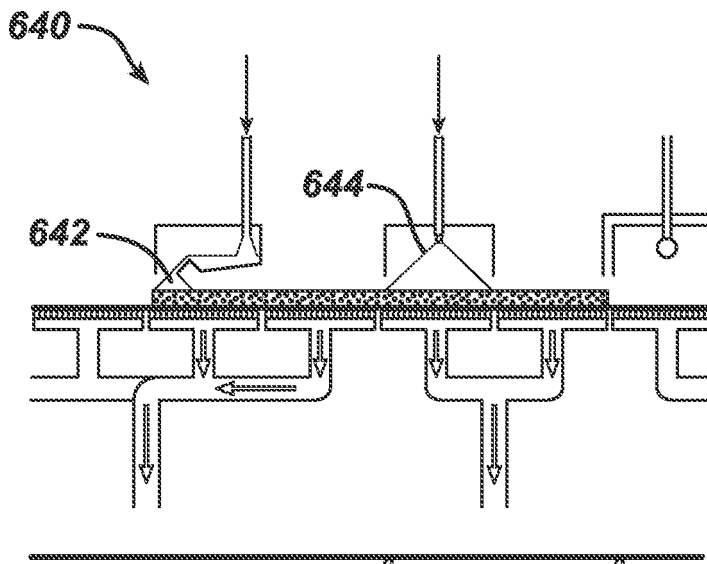


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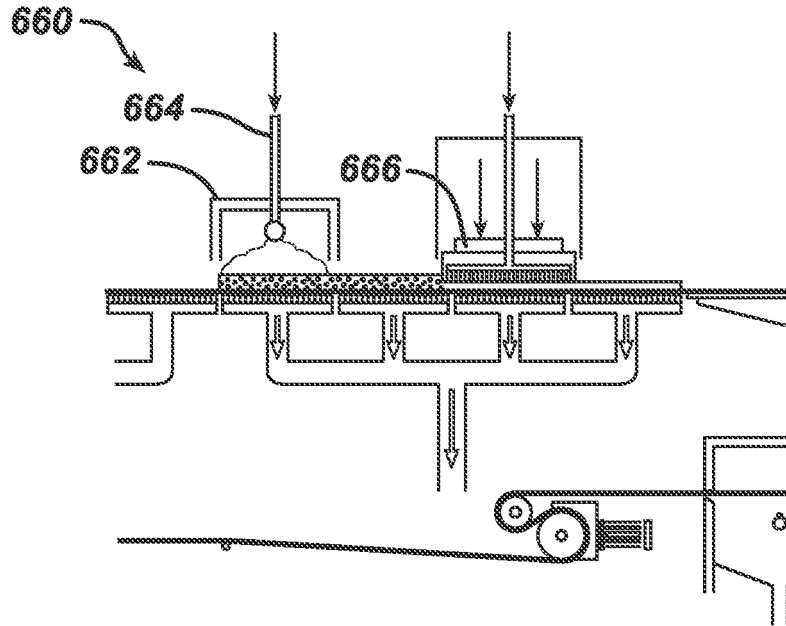
**FIG. 5B**



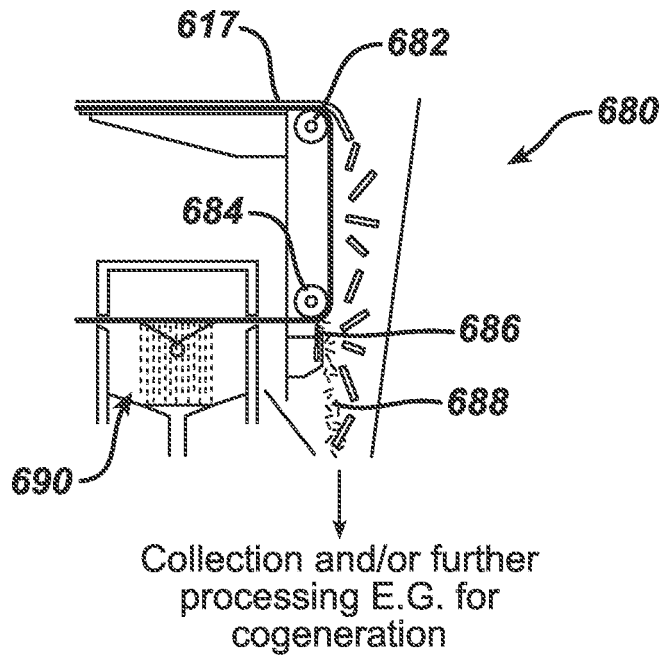
**FIG. 5C**



**FIG. 5D**



**FIG. 5E**



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