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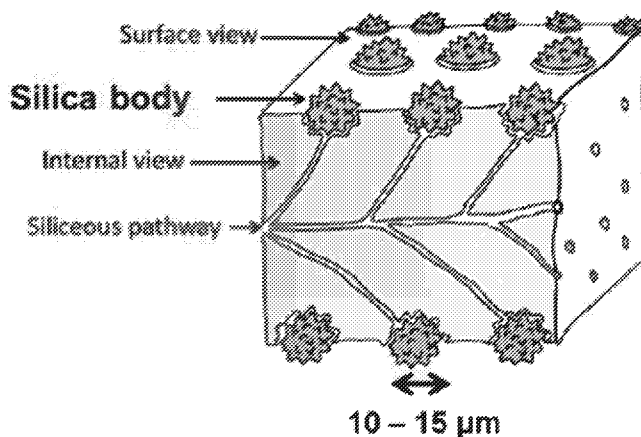
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(54) Title: PROCESS FOR TREATING NON-WOOD FEEDSTOCK

FIG. 1



(57) Abstract: Methods for pre-treating non-wood feedstock, in particular, empty fruit bunch (EFB) 5 feedstock and pulps obtained using these methods are described.



Process for Treating Non-Wood Feedstock

The present invention provides methods for pre-treating non-wood feedstock, in particular, empty fruit bunch (EFB) feedstock, a waste product formed after the extraction of palm oil, wherein the pre-treated non-wood feedstock is for subsequent use in producing paper or textile grade pulps for application in industries such as printing writing paper, tissue paper, paper board and fashion garments.

Background

An increased emphasis on environmental sustainability, coupled with a global shortage of wood feedstock for the pulp and paper industries has meant that focus has shifted towards finding alternative, non-wood feedstock sources. The same is true for the textiles industry where there is a drive towards using renewable raw materials in the production of fibers.

Examples of non-wood feedstocks which provide interesting replacements to wood feedstocks include oil palm empty fruit bunch (EFB) fibers, bamboo, kenaf, wheat/rice straw, coconut coil and bagasse.

Oil palm (*elaeis guineensis*), which is typically grown in Southeast Asian countries such as Indonesia, Malaysia and Thailand, is an important source of vegetable oil, specifically palm oil. Various biomass is produced during the extraction of palm oil. One of the most abundant biomasses produced is Empty Fruit Bunches (EFB) fibers, produced by the first step in the oil extraction process wherein the fruit and nuts are stripped from the fruit bunches. EFB generation is increasing year on year and in 2015 alone, over 30 million tonnes was produced in Indonesia alone. This, in itself, presents an environmental issue as often this waste is simply left to rot and attract rodents.

EFB has a number of properties which make it of interest as a raw material for pulp – it has a high cellulose fiber content and a low lignin content, it resembles hardwood fibers and can be used to provide paper and regenerated cellulose of a high quality. Processes are known in which EFB is used as a raw material which is then subjected to cooking in order to produce chemical pulps from which it is then possible to produce paper and regenerated cellulose, e.g. viscose, modal, lyocell and other man-made cellulose (MMC) fibers.

While EFB and other non-woods are promising replacements for wood in both the paper and textile industries, an issue which arises with these feedstocks is the presence of impurities which cause issues with either processing and/or the quality of the end product. This means

that where EFB and other non-woods are used as a feedstock the pulping process needs to be adapted to include pre-treatment steps to remove these impurities. Impurities which are particularly problematic include oils (in the form of fatty acids), silica, ash and other non-process elements (NPEs) content. Although it has been possible to develop processes for the effective removal of oils, ash residue and NPEs, the removal of silica has been found to be difficult.

While difficult with non-wood feedstocks in general, the removal of silica from EFB feedstock is particularly difficult. Investigation of EFB has revealed that it has a particular structure which consists of silica bodies embedded in fiber (see Figure 1) and it is believed that it is these silica bodies which contribute towards the strength and rigidity of EFB (see also Law *et al.*, (2007) *Bioresources* 2(3), 351-362). However, as described above, the presence of silica in a pulp mill is undesirable for a number of reasons, specifically issues with recovery, poor product quality and damage to machinery as a consequence of silica deposition during processing. Therefore, methods for pre-treating EFB feedstock to remove silica have been investigated. To date, pre-treatment methods have included physical, chemical and biological treatments, for example, washing with water, treatment with acid, ultrasonic treatment, fungal/enzyme treatment and treatment with alkali (see Agusta *et al.*, (2018) *Journal of Japan Tappo* 6, 641-49; Tye *et al.* (2013) *Procedia Environmental Sciences* 20, 328-335; Farah *et al.*, 2014, *BioResource* 9(1), 938-951; Nik *et al.*, (2008), *MCRJ Special Issue* 4(2), 117-128; Zawati *et al.*, (2015) *Wood Research* 60(1), 157-166; Harsono *et al.*, (2015), *Journal of Wood Science* 62, 55-73; Rusdan (2002) *Oil Palm Bulletin* 44, 19-24); Rusdan *et al.*, (2018) *Journal of Tropical Forest Science* 19(3), 121-126; ; Bahrin *et al.*, (2012) *Bioresources* 7(2), 1784-1801; and Isroi *et al.*, *Molecules* 17(12), 14995-15002).

With any pre-treatment process, it is necessary to balance the removal of the impurity, in this case, silica bodies, against the damage caused to the EFB feedstock by the pre-treatment process. The majority of these pre-treatments have been described in the context of a producing either sugar for bioethanol production, the production of furfural or the production of chemical pulps. In contrast, the present invention is primarily concerned with processes for producing man-made cellulosic fibers (regenerated cellulose) for the textiles industry, such as viscose, which are particularly sensitive to both the presence of silica and any fiber damage incurred during earlier processing steps. Thus, while the known processes may remove enough silica for the end applications for which they have been designed, the levels of silica which remain are still too high to be acceptable in the regenerated cellulose fiber textiles industry.

Therefore, it is clear that there is a need for pre-treatment processes for non-wood feedstock which is ultimately to be used in fiber manufacture, which remove high levels of silica without damaging the non-wood feedstock.

5 Summary of invention

Against this background, the present inventors have identified pre-treatment processes which are effective in removing silica bodies from non-wood feedstock, while avoiding causing significant damage to the cellulosic component of the feedstock.

10 More specifically, the present invention provides a method for pre-treating non-wood feedstock comprising:

- (a) washing non-wood feedstock;
- (b) contacting the washed non-wood feedstock with an acid solution; and
- (c) contacting the washed non-wood feedstock with an alkali solution;

15 wherein the method includes an additional washing step (d) between steps (b) and (c).

Advantageously, the inventors have found that by carrying out the steps (a), (b), (c) and (d) this pre-treatment process results in a feedstock which contains very little silica while retaining its structural integrity and minimising damage to the cellulosic component of the feedstock. In particular, the inventors have surprisingly found that by combining a series of steps, each of which is carried out under conditions which are sufficiently mild to avoid unwanted structural damage, it is possible to remove effectively problematic silica bodies from the fibers of the feedstock. This is particularly surprising given the previous understanding in the industry that extreme and/or harsh conditions are required in order to remove these silica bodies.

25 Furthermore, the pre-treatment method of the present invention can be incorporated easily into existing saw dust/pin chip mills, organosolv and soda or kraft pulp mills. It is particularly advantageous for use in an integrated pulp mill *i.e* a mill in which the complete process from feedstock to end product is performed, because reactants from later stages in the process can be recycled back into the pre-treatment method, in particular step (b). This improves efficiency and minimises waste.

35 The present invention further provides Dissolving Pulp (DP) derived from non-wood feedstock, in particular non-wood empty fruit bunch (EFB) feedstock, which has a silica content of less than about 80 ppm. Advantageously, the low levels of silica present in the DP mean that it can be subsequently processed into regenerated cellulose fibers for use in the textiles industry to produce a high quality product. In particular, where silica is still present in the DP, it will cause

issues with cellulose dissolution and clog the spinnerets which are subsequently used to draw the DP into fibers.

5 The present invention yet further provides Kraft Pulp (KP) derived from non-wood feedstock, in particular non-wood empty fruit bunch (EFB) feedstock, which has a silica content of less than about 600 ppm. Advantageously, the low levels of silica present in the KP mean that it can be subsequently processed into paper products without damaging the pulping machinery used. In particular, where silica is still present in the KP, it can lead to its deposition in the pulping machinery (*i.e.* sand deposits) which, as well as potentially damaging the machinery, can also
10 lead to holes in the end product. The KP produced by the methods described herein does not suffer from these problems.

The inventors have found that the DP and KP obtained from feedstock which has been pre-treated according to the methods of the present invention has a quality which is equivalent to or
15 better than that obtained using conventional wood feedstocks. It can also be obtained in a comparable yield.

Detailed Description

The pre-treatment method of the present invention can be used with a variety of non-wood
20 feedstocks. In this regard, the term “non-wood feedstock” is used herein to describe any feedstock which is plant-based but which has not been derived from a wood source. Examples of suitable non-wood feedstock include oil palm empty fruit bunches (EFB), bamboo, bagasse, kenaf, coconut coil and wheat/rice straw. Preferably the non-wood feedstock is EFB feedstock or bamboo feedstock. Most preferably, the non-wood feedstock is EFB feedstock.

25 The term “Non-Process Element (NPE)” is used herein to refer to unwanted inorganic impurities other than silica and ash, such as metals including iron and calcium.

The first step in the pre-treatment process of the present invention is step (a) in which the non-
30 wood feedstock is washed. This washing step is included first for the purpose of removing residual oils from the non-wood feedstock. Washing may be carried out using either water and/or steam. Preferably washing step (a) is performed at a temperature in the range from 80°C to 100°C. Where water is used in washing step (a), at the lower end of this temperature range, it will be appreciated that a mixture of water and steam is present, while at the upper end of the
35 temperature range, steam is present. Preferably, the non-wood feedstock is mechanically agitated, preferably by gentle mechanical agitation, during step (a) of the method of the present invention. Preferably, the non-wood feedstock is agitated only to a small degree, such as that

which may be achieved by passing the non-wood feedstock through a screw-type conveyor. The skilled person will be familiar with screw-type conveyors, an example of which is shown in Figure 11.

- 5 Prior to step (a) of the method of the present invention, the non-wood feedstock may have been subjected to process steps which are standard in the industry, for example shredding. Where the non-wood feedstock is shredded, preferably the resulting fibers have a length in the range from about 5 mm to about 100mm.
- 10 After the initial washing step (a), the washed non-wood feedstock is contacted with an acid solution in step (b) of the method. Contacting the non-wood feedstock with acid reduces the content of any residual metal impurities. In particular, it is effective in removing iron and calcium residues from the feedstock. Furthermore, without wishing to be bound by theory, it is believed that this acid treatment step is also crucial in the process of removing silica from the feedstock.
- 15 As described previously, non-wood feedstock, in particular EFB feedstock, has a structure in which silica bodies are embedded in the surfaces of the fibers. Contacting the fibers with acid causes the fibers to shrink, which in turn, starts a process of displacing the silica bodies.

- The acid solution comprises an acid and water. Any acid may be used to form the acid solution.
- 20 Preferably the acid used is selected from the group consisting of sulfuric acid, hydrochloric acid, a solution of chlorine dioxide in water and mixtures thereof. As will be described later, a pulp mill typically includes a bleaching step. An advantage of the pre-treatment method of the present invention is that the acid used in step (b) of the pre-treatment method can be provided by acid used in a bleaching step being recycled directly into step (b) of the pre-treatment
- 25 method, thus improving efficiency, minimising waste and cost production.

- The acid is included in the acid solution of step (b) at a concentration in the range from about 0.2 to 5.0%. This ensures that the acid solution is sufficiently concentrated that the fibers of the non-wood feedstock shrink to facilitate removal of silica impurities but is dilute enough that
- 30 unwanted damage to the feedstock, in particular to the cellulose in the feedstock, is minimised.

Preferably step (b) is carried out at pH in the range from 1 to 3.

- Step (b) is generally carried out at a temperature in the range from about 80°C to about 100°C.
- 35 It is preferable to perform step (b) at an elevated temperature because it increases the speed with which step (b) proceeds and so the efficiency of the process.

Where the non-wood feedstock from step (a) has been washed at an elevated temperature as described previously, also performing step (b) at an elevated temperature (*i.e.* a temperature in the range from about 80 to about 100°) improves efficiencies in the overall process as it avoids the need to include a step before step (b) in which the washed non-wood feed stock is cooled.

5

Step (b) is carried out for a time sufficient to achieve shrinkage of the fibers. Preferably, step (b) has a duration in the range from about 30 to about 60 minutes. The extent to which step (b) has proceeded can be monitored by measuring the content of the impurities which are to be removed, specifically ash residue, NPEs and silica. At the end of step (b), typically the silica content will have reduced by a small amount, while ash residue and NPEs will have been almost completely removed. A person skilled in the art will be familiar with suitable quality measurements.

In step (c) of the method of the present invention, the washed non-wood feedstock is contacted with an alkali solution. Contacting the feedstock with an alkali solution further assists in the removal of silica impurities. More specifically, contacting the feedstock with an alkali solution causes the fibers to swell which contributes towards removal of the silica from the feedstock.

Step (c) is carried out after step (a) and may be performed either before or after step (b). In one method according to the present invention, step (c) is performed before step (b). In an alternative method according to the present invention, step (c) is performed after step (b). Preferably step (c) is performed after step (b). This is advantageous because, as will be described in more detail later, after pre-treatment, the non-wood feedstock is cooked to form a pulp. A preferred cooking step is performed under alkaline conditions and hence, if the pre-treatment method finishes with an alkaline step, it is already at a pH which is appropriate for the cooking step and thus the need for an additional neutralization step is avoided.

It is the combination of steps (b) and (c), which shrink and swell the fibers of the non-wood feedstock, respectively, which leads to effective removal of silica impurities.

30

The alkali solution used in step (c) comprises an alkali and water. Any alkali may be used to form the alkali solution but preferably the alkali is sodium hydroxide. Sodium hydroxide is preferred because, as described above, it is also used in the later cooking step described above directly after pre-treatment. By using the same alkali in step (c), the overall process efficiency is improved.

35

The alkali is included in the alkali solution of step (c) at a concentration in the range from about 0.2 to about 5.0%. This ensures that the alkali solution is sufficiently concentrated that the fibers of the non-wood feedstock swell to facilitate removal of silica impurities but is dilute enough that unwanted damage to the feedstock, in particular cellulose in the feedstock, is minimised.

5

Preferably step (c) is carried out at a pH in the range from 12 to 14.

Step (c) is generally carried out at a temperature in the range from about 80°C to about 100°C. It is preferable to perform step (c) at an elevated temperature because it increases the speed with which step (b) proceeds and so the efficiency of the process.

10

Where the non-wood feedstock from step (a) has been washed at an elevated temperature as described previously, also performing step (c) at an elevated temperature (*i.e.* a temperature in the range from about 80 to about 100°) improves efficiencies in the overall process as it avoids the need to include a step before step (c) in which the washed non-wood feed stock is cooled.

15

Step (c) is carried out for a time sufficient to achieve swelling of the fibers. Preferably, step (c) has a duration in the range from about 30 to about 60 minutes. The extent to which step (c) has proceeded can be monitored by measuring the content of silica which remains in the feedstock. At the end of step (c), typically the silica content will have reduced significantly. A person skilled in the art will be familiar with suitable quality measurements.

20

The pre-treatment method of the present invention includes a further washing step, step (d), between steps (b) and (c), irrespective of the order in which steps (b) and (c) are performed.

25

This washing step is included for the primary purpose of removing metal and silica (typically fine silica) impurities which have been displaced from the non-wood fibers as a consequence of the earlier steps which have been performed. The washing of step (d) may be carried out using either water and/or steam. Preferably washing step (d) is performed at a temperature in the range from 80°C to 100°C. Where water is used in washing step (d), at the lower end of this temperature range, it will be appreciated that a mixture of water and steam is present, while at the upper end of the temperature range, steam is present. Preferably, the non-wood feedstock is mechanically agitated, preferably by gentle mechanical agitation, during step (a) of the method of the present invention. Preferably, the non-wood feedstock is agitated only to a small degree, such as that which may be achieved by passing the non-wood feedstock through a screw-type conveyor. The skilled person will be familiar with screw-type conveyors, an example of which is shown in Figure 11.

30

35

The method of the present invention may include a further washing step (e) after whichever of step (b) or step (c) is performed last. Where present, this additional washing step is included for the primary purpose of removing silica impurities. The washing of step (e) may be carried out using either water and/or steam. Preferably washing step (e) is performed at a temperature in the range from 80°C to 100°C. Where water is used in washing step (e), at the lower end of this temperature range, it will be appreciated that a mixture of water and steam is present, while at the upper end of the temperature range, steam is present. Preferably, the non-wood feedstock is mechanically agitated, preferably by gentle mechanical agitation, during step (a) of the method of the present invention. Preferably, the non-wood feedstock is agitated only to a small degree, such as that which may be achieved by passing the non-wood feedstock through a screw-type conveyor. The skilled person will be familiar with screw-type conveyors, an example of which is shown in Figure 11.

Once subjected to the pre-treatment method of the present invention, the non-wood feedstock is suitable for further processing to form a pulp which can then, in turn, serve as a raw material for either paper or in the production of regenerated cellulose, for example viscose, modal, lyocell or other MMC fibers.

The methods of the present invention can be carried out using standard apparatus found in standard pulp mills with which the person skilled in the art will be familiar. In particular, the pre-treatment method of the present invention is useful in an integrated pulp mill so that materials *e.g.* acid which is used in a bleaching step can be recycled into the pre-treatment methods of the present inventions.

Prior to further processing, the non-wood feedstock may be formed into bales. Alternatively, prior to further processing, the non-wood feedstock may be pelletized.

A person skilled in the art will be familiar with suitable methods for converting the pre-treated non-wood feedstock into pulp. In particular, there are two general methods which are used for cooking the feedstock, specifically a sulfate (Kraft) process and a pre-hydrolysis (PH) kraft process.

In a Kraft process, the pre-treated non-wood feedstock is mixed with a hot mixture, known as "white liquor" which comprises water, sodium hydroxide and sodium sulfite. Anthraquinone may be included as an accelerant to improve yields. During this process, lignin present in the feedstock are broken down and dissolved in the alkali. The remaining solid pulp, known as "brown stock" can be collected ready to be subjected to further processing steps. The combined

liquids (the “black liquor”) which contain lignin fragments, a small fraction of carbohydrates, sodium sulfate, sodium carbonate and other inorganic salts, can be removed and then burned to provide energy which is fed back into the cooking process.

- 5 The “brown pulp” obtained in the Kraft process is typically bleached in order to provide a pulp which has a high brightness. There are a number of different bleaching processes which may be used and the skilled person will be familiar with these. An example of a suitable bleaching process is Elemental Chlorine Free (ECF) bleaching in which the brown pulp is contacted sequentially with peroxymonosulfuric acid (which can be produced by mixing sulfuric acid and
- 10 hydrogen peroxide), chlorine dioxide, hydrogen peroxide and chlorine dioxide. Another example of a suitable bleaching process is Total Chlorine Free (TCF) bleaching in which the brown pulp is subjected to bleaching with ozone and hydrogen peroxide. In particular, TCF typically includes four or five different stages including both treatment with chelating agent and several bleaching stages such as bleaching with oxygen, alkaline extraction with hydrogen peroxide
- 15 addition, peracetic acid bleaching and/or hydrogen peroxide bleaching with or without oxygen. TCF bleaching has the advantage that no chlorine is used and so no chlorine-containing byproducts are generated. The quality of the pre-treated non-wood feedstock obtained by the method of the present invention means that less energy is required in the bleaching step.
- 20 An advantage of the pre-treatment method of the present invention is that the acid used in such a bleaching step can be recycled back into step (b) of the pre-treatment method, thus reducing the amount of acid required for the overall process and reducing waste.

In a PH kraft process, the non-wood feedstock is first subjected to a water-based auto-

25 hydrolysis process which removes hemicelluloses and some lignin. It is then cooked (kraft cooking) under alkaline conditions to remove the majority of lignin and a small fraction of remaining hemicellulose. Final multi-stage bleaching steps (e.g. ECF bleaching or TCF bleaching) are performed to increase the final pulp brightness. Anthraquinone can be included as an accelerant to improve yields in a PH kraft process.

30 Preferably the pre-treated non-wood feedstock obtained from the method of the present invention is cooked using a PH Kraft process.

In both cooking methods, the quality of the pulp which is obtained can be controlled by varying

35 several parameters, specifically the H-factor, the P-factor, the Kappa number and Active Alkali%. In particular, by controlling these parameters, it is possible to control whether the pulp obtained is Dissolving Pulp (DP) or Kraft Pulp (KP).

Dissolving Pulp (DP) is a high grade pure cellulosic pulp which has a high level of brightness, a uniform molecular weight distribution and an open pore structure, rendering it highly reactive. DP is typically used in the production of regenerated cellulose.

5

Kraft Pulp (KP) is a pulp which contains both cellulose and hemicellulose. KP is typically used in the production of paper.

10

As will be appreciated by the person skilled in the art, these parameters have standard definitions. The H-factor is a kinetic model for the rate of delignification and depends on both temperature and time. The P-factor depends on the time and the temperature in a water-based medium. The Kappa number is a measure of the completeness of the process and gives an indication of the amount of lignin which remains in the pulp; it is measured as the amount of standard potassium permanganate solution that the pulp will consume. The Active Alkali % is the total concentration of alkaline constituents, except carbonates, present in the reaction as determined by titration of a sample of the white liquor with strong acid according to the procedure which is set out in SCAN-N-30.

15

Examples of suitable parameter ranges to obtain DP and KP are set out in Table 1 below.

20

Table 1

Parameter	KP	DP
Active Alkali (AA)%	15 to 20	15 to 20
P factor (150-170°C) [£]	NA	1000-1300
H factor (150 to 170°C) [£]	100 to 300	100 to 300
Kappa*	14 – 20	6 -10

*measured according to Tappi T236-om-06.

[£]measured using standard digester computer software with which the skilled person will be familiar.

25

As a consequence of the pre-treatment method of the present invention to which the non-wood feedstock has been subjected, the present invention provides DP and KP which has a very low impurity content.

30

It has also been found that, surprisingly, where a non-wood feedstock is treated according to the methods of the present invention prior to cooking, shorter cooking times are required. This means that less energy is required for the cooking process. It has further been found that non-

wood feedstock treated according to the methods of the present invention requires less bleaching. In addition, as can be seen in Figure 10, in the subsequent bleaching of non-wood feedstock which has been treated according to the methods of the present invention, there is a significant reduction in the bleaching chemicals required, particularly where the non-wood feedstock is EFB feedstock where the consumption of chemicals is almost halved.

In particular, the DP and KP obtained using the pre-treatment method of the present invention meets the specifications as set out in Table 2 below.

Table 2

Parameter	KP	DP
Silica / ppm	400 to 600	<80
Fe / ppm	No specification	<10
Ca / ppm	No specification	<75
Ash / %	<0.35	< 0.12
Brightness (ISO) / %**	89.5	90
Viscosity / ml/g***	450 – 900	350 - 550

**Measured according to Tappi T525-om-06

***Measured according to Tappi T230-om-04 at 25°C

The low silica content of the DP obtained using the methods of the present invention is particularly surprising given the previous issues which have been encountered in attempts to remove silica from a non-wood feedstock. This is particularly the case where the non-wood feedstock is EFB feedstock.

In this regard, the present invention provides dissolving pulp derived from non-wood feedstock which has a silica content of less than about 80 ppm.

Silica content can be determined according to the method set out in standard Tappi T 211-om-07.

Alternatively or in addition, the dissolving pulp derived from non-wood feedstock has a Fe content of about 10ppm or less.

Iron (Fe) content can be determined according to the method set out in standard Tappi T618-om-01.

Alternatively or in addition, the dissolving pulp derived from non-wood feedstock has a Ca content of about 75 ppm or less.

Calcium content can be determined according to the method set out in standard Tappi T618-
5 cm-01.

The presence of Fe and/or Ca impurities within the DP is undesirable because it affects subsequent steps in which cellulose is dissolved and ultimately leads to the need to increase the amounts of chemicals where are used in such steps.

Alternatively or in addition, the dissolving pulp derived from non-wood feedstock has an ash content of about 0.12% by weight or less.

The ash content can be determined according to the method set out in standard Tappi T322-
15 om-07.

The present invention further provides kraft pulp derived from non-wood feedstock which has a silica content of less than about 600 ppm.

Alternatively or in addition, the kraft pulp derived from non-wood feedstock has an ash content of about 0.35% or less.

In particular, the present invention provides dissolving pulp derived from non-wood empty fruit bunch (EFB) which has a silica content of less than about 80 ppm.

Alternatively or in addition, the dissolving pulp derived from non-wood EFB has a Fe content of about 10ppm or less.

Alternatively or in addition, the dissolving pulp derived from non-wood EFB has a Ca content of about 75 ppm or less.

Alternatively or in addition, the dissolving pulp derived from non-wood EFB has an ash content of about 0.12% or less.

The present invention further provides kraft pulp derived from non-wood empty fruit bunch (EFB) which has a silica content of less than about 600 ppm.

Alternatively or in addition, the kraft pulp derived from non-wood empty fruit bunch (EFB) has an ash content of about 0.35% or less.

5 The DP of the present invention has a high brightness. Brightness is a measure of the amount of incident light reflected from the pulp under specified conditions as measured by the method set out in standard Tappi T525-om-06. The DP of the present invention preferably has a Brightness (ISO) of about 90% or greater.

10 The KP of the present invention has a high brightness. Brightness is a measure of the amount of incident light reflected from the pulp under specified conditions as measured by the method set out in standard Tappi T525-om-06. The KP of the present invention preferably has a Brightness (ISO) of about 90% or greater.

15 The dissolving pulp obtained by the methods of the present invention may be further processed to provide regenerated cellulose. The term "regenerated cellulose" is used herein to describe a class of materials manufactured by the conversion of natural cellulose from a non-wood feedstock, to a soluble cellulosic derivative and subsequent regeneration, typically forming either a fiber or a film. A person skilled in the art will be familiar with techniques for processing dissolving pulp to form regenerated cellulose. A first step in this further processing is typically to
20 convert the dissolving pulp into cellulose dope. Where the end product is viscose textile fibers, in a next step, the cellulose dope is typically subjected to a xanthation in the presence of CS₂ and sodium hydroxide. Where the end product is Lyocell textile fibers, the cellulose dope is typically subjected to a N-methylmorpholine N-oxide (NMMO) reaction. In both cases, the product of the subsequent step can be extruded through spinnerets and regenerated to form the
25 desired man-made fibers, *e.g.* viscose and/or lyocell textile fibers. It is also possible to produce other man-made cellulose (MMC) fibers. It is an attractive option for the textiles industry because it can be produced using significantly less water than *e.g.* cotton and the resultant material is highly breathable.

30 The kraft pulp obtained by the methods of the present invention may be further processed to provide paper products. The person skilled in the art will be familiar with techniques for forming a paper product from pulp. In particular, processes typically involve suspending the pulp in water and then machinery to flatten, dry and cut to form sheets and rolls.

35 The invention will now be further described by reference to the following figures and examples which are in no way intended to be limiting on the scope of the claims.

Figures

Figure 1 is an illustration of the position of silica bodies within an EFB fiber;

Figure 2 is a block diagram illustrating a pre-treatment method of the present invention;

Figure 3(a) shows the silica content of non-wood EFB feedstock before and after the pre-treatment according to the method of the present invention and as compared to prior art techniques;

Figure 3(b) shows the silica content of non-wood bamboo feedstock before and after the pre-treatment according to the method of the present invention and as compared to prior art techniques;

Figure 4(a) shows the ash content of non-wood EFB feedstock before and after pre-treatment according to the method of the present invention and as compared to prior art techniques;

Figure 4(b) shows the ash content of non-wood bamboo feedstock before and after pre-treatment according to the method of the present invention and as compared to prior art techniques;

Figure 5 shows the silica contents of a non-wood EFB feedstock and a non-wood bamboo feedstock before and after the pre-treatment methods of the present invention;

Figure 6 is an image of non-wood EFB KP produced by the method of the present invention;

Figure 7 is an image of non-wood EFB DP produced by the method of the present invention;

Figure 8 is a block diagram illustrating a process for producing paper products which includes the pre-treatment method of the present invention; and

Figures 9(a) is a block diagram illustrating a process for producing viscose which includes the pre-treatment of the present invention;

Figure 9(b) is a block diagram illustrating a process for producing lyocell which includes the pre-treatment of the present invention; and

Figure 10 shows the consumption of chlorine dioxide (ClO_2) during an ECF bleaching process performed on EFB feedstock treated according to the methods of the present invention as compared to hardwoods, specifically acacia crassicarpa (ACRA), Eucalyptus pellita (EPEL), Eucalyptus hybrid (EHYB);

Figure 11 shows a screw-type conveyor which may be used to move the non-wood feedstock from one stage to another in the method of the present invention;

Figure 12 shows the dates and times at which the feedstock was changed from a standard wood feedstock to a mixed EFB/wood feedstock in Example 3;

Figure 13 illustrates the productivity (H-factor) of the mill where the feedstock is changed from wood chip to mixed EFB/wood chip during the trial described in Example 3;

Figure 14 shows the oxygen charge consumed during bleaching in the mill described in Example 3;

Figure 15 shows the alkali charge consumed during bleaching in the mill described in Example 3;

Figure 16 shows the silica content of bleached kraft pulps obtained from the mixed feedstock as described in Example 3; and

- 5 Figure 17 illustrates the dirt spot/count of bleached kraft pulps obtained from the mixed feedstock as described in Example 3.

Examples

Example 1 – non-wood EFB feedstock

10

Approximately 100kg of non-wood EFB which had been processed so that it was free from kernel, palm seed and mud was obtained from Asian Agri (RGE palm oil business) and was passed into a shredder and washer to obtain EFB feedstock having a fiber length in the range from 5 to 100mm.

15

The silica and ash residue contents of the non-wood EFB were measured according to Tappi T618-cm-01. The results are shown in Figures 3(a) and 4(a).

20

The non-wood EFB feedstock was then washed using a mixture of water and steam at a temperature in the range from 80 to 100°C in a drum washer. The EFB feedstock was moved towards the next treatment step by screw conveyor (which provides a small degree of mechanical agitation). The washing step was carried out for a duration of 30 to 60 mins. The washed EFB feedstock was then passed to a vessel which contained acid solution in sufficient liquor ratio (7 to 10) having a pH in the range from 1 to 3 and at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the EFB feedstock was removed from the acid-containing vessel and passed to a drum washer where it was washed using a mixture of water and steam at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the washed and acid-treated EFB feedstock was passed to a vessel which contained alkali solution in sufficient liquor ratio (7 to 10) having a pH in the range from 12 to 14 and at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the EFB was removed from the alkali-containing vessel and passed to a drum washer where it was washed using a mixture of water and steam at a temperature in the range from 80 to 100°C. The pre-treated EFB feedstock was moved toward the next step by a screw conveyor (which provides a small degree of mechanical agitation).

25

30

35

The silica and ash residue contents of the pre-treated EFB feedstock were measured according to Tappi T618-cm-01 and are illustrated in Figures 3(a) and 4(b).

The pre-treated EFB feedstock was then cooked under prehydrolysis kraft (P- factor 1000 to 1300)/kraft for both DP and KP. Active alkali was used in an amount in the range from 15 to 20% and the H factor was in the range from about 100 to 300 (see Table 1). The target specifications for chemical pulps (DP & KP), as set out in Table 2 were achieved.

5

Example 2 – non-wood bamboo feedstock

Approximately 5kg of non-wood bamboo bark was chipped and passed through a mesh screen of size 7 to 13 mm to form bamboo chips of a standard size-

10

The silica and ash residue contents of the non-wood bamboo were measured according to Tappi T618-cm-01. The results are shown in Figures 3(b) and 4(b).

15

The non-wood bamboo feedstock was then washed using a mixture of water and steam at a temperature in the range from 80 to 100°C in a drum washer. The bamboo feedstock was moved towards the next treatment step by a conveyor belt. The washing step was carried out for a duration of 30 to 60 mins. The washed bamboo feedstock was then passed to a vessel which contained acid solution in sufficient liquor ratio (4 to 7) having a pH in the range from 1 to 3 and at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the bamboo feedstock was removed from the acid-containing vessel and passed to a drum washer where it was washed using a mixture of water and steam at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the washed and acid-treated bamboo feedstock was passed to a vessel which contained alkali solution in sufficient liquor ratio (4 to 7) having a pH in the range from 12 to 14 and at a temperature in the range from 80 to 100°C. After 30 to 60 mins, the bamboo feedstock was removed from the alkali-containing vessel and passed to a drum washer where it was washed using a mixture of water and steam at a temperature in the range from 80 to 100°C. The pre-treated bamboo feedstock was moved toward the next step by a conveyor belt.

20

25

The silica and ash residue contents of the pre-treated bamboo feedstock were measured according to Tappi T618-cm-01 and are illustrated in Figures 3(b) and 4(b).

30

The pre-treated bamboo feedstock was then cooked under prehydrolysis kraft (P- factor 500 to 800)/ kraft for both DP and KP. Active alkali was used in an amount in the range from 15 to 20 % and the H factor was in the range from about 500 to 700. The target specification for chemical pulps (DP & KP), as set out in Table 2 earlier was achieved.

35

Example 3 - EFB feedstock Mill trial

Standard wood feedstock was replaced with a mixed wood feedstock comprising 5 to 10% by weight EFB feedstock in a running mill which processes 35 tonnes of feedstock per day.

- 5 Various outputs were monitored as summarised below. The process to which the mixed EFB feedstock was subjected included the pre-treatment method of the present invention.

Figure 12 illustrates the point at which the feedstock was altered to comprise 5 to 10% by weight of EFB feedstock. It is clear from Figure 13 that a higher cooking productivity (as evidenced by a lower H-factor) was observed for the same level of alkali charge and kappa number where the feed was changed to a mixed EFB feedstock. With reference to Figures 14 and 15, it is also clear that the consumption of chemicals in the bleaching step was reduced – both the oxygen and alkali charges were reduced and the total charge of ClO_2 was also reduced (47.3 kg for pure wood feedstock vs. 46.2kg for mixed EFB/wood feedstock).

15 In addition to these reductions in both chemical consumption and energy required, advantageously, it has been found that the quality of the final bleached pulps were maintained. In particular, the pulps obtained had the following properties:

20 Table 3

Parameter	Value
Brightness	89.5%
Viscosity	631 dm^3/kg
Freeness	536 ml CSF
Dirt	$6\text{mm}^2/\text{m}^2$
Silica content	432 mg/l

The end product in the mill trial described in Example 3 was a paper product. Where the feedstock was changed from pure wood to a mixture of EFB and wood and the process included the pre-treatment method of the present invention, it was surprisingly found that the end paper product had excellent properties, specifically a tensile strength of 70 Nm/g and a bulk density of $1.39\text{ cm}^3/\text{g}$. The paper also demonstrated high performance in both drainability and runnability.

Overall, in replacing the wood feedstock with a mixed EFB/wood feedstock and including the pre-treatment method of the present invention, it was possible to produce products having the required excellent properties while reducing energy consumption by approximately 10%.

Exemplary Embodiments

The present invention is further described by reference to the following numbered exemplary
5 embodiments.

1. A method for pre-treating non-wood feedstock comprising:
 - (a) washing non-wood feedstock;
 - (b) contacting the washed non-wood feedstock with an acid solution; and
 - 10 (c) contacting the washed non-wood feedstock with an alkali solution;wherein the method includes an additional washing step (d) between steps (b) and (c).
2. A method according to embodiment 1, wherein step (b) is performed before step (c).
- 15 3. A method according to embodiment 1, wherein step (c) is performed before step (b).
4. A method according to any preceding embodiment, wherein in step (a), the non-wood feedstock is washed using water and/or steam.
- 20 5. A method according to any preceding embodiment, wherein step (a) is performed at a temperature in the range from 80 to 100°C.
6. A method according to any preceding embodiment, wherein in step (a), the non-wood feedstock is passed through a screw-type conveyor.
- 25 7. A method according to any preceding embodiment, wherein in step (b), the washed non-wood feedstock is contacted with the acid solution at a temperature in the range from 80 to 100°C for a duration of 30 to 60 minutes.
- 30 8. A method according to any preceding embodiment, wherein in step (b), the pH is in the range from 1 to 3.
9. A method according to any preceding embodiment, wherein in step (b) the acid solution comprises an acid selected from the group consisting of sulfuric acid, hydrochloric acid,
35 a solution of chlorine dioxide in water and mixtures thereof.
10. A method according to any preceding embodiment, wherein in step (b) the acid solution comprises acid at a concentration of about 0.2 to about 5%.

11. A method according to any of embodiments 1 to 9, wherein in step (b) the acid solution comprises an acid solution which has been recycled from a step in which dissolving pulp (DP) and/or kraft pulp (KP) has been bleached.
- 5
12. A method according to embodiment 11, wherein in step (b) the acid solution comprises recycled acid at a concentration of about 0.2 to about 5%.
13. A method according to any preceding embodiment, wherein in step (d), the non-wood
- 10 feedstock is washed using water and/or steam.
14. A method according to any preceding embodiment, wherein step (d) is performed at a temperature in the range from 80 to 100°C.
- 15
15. A method according to any preceding embodiment, wherein in step (d), the non-wood feedstock is passed through a screw-type conveyor.
16. A method according to any preceding embodiment, wherein in step (c), the washed non-wood feedstock is contacted with the alkali solution at a temperature in the range from
- 20 80 to 100°C for a duration of 30 to 60 minutes.
17. A method according to any preceding embodiment, wherein in step (c), the pH is in the range from 12 to 14.
- 25
18. A method according to any preceding embodiment, wherein in step (c), the alkali solution comprises sodium hydroxide.
19. A method according to embodiment 18, wherein in step (c), the alkali solution comprises sodium hydroxide at a concentration of about 0.2 to about 5%.
- 30
20. A method according to any preceding embodiment which further comprises:
(e) washing the product obtained after steps (a) to (d).
21. A method according to embodiment 20, wherein in step (e), the product is washed with
- 35 water and/or steam.

22. A method according to embodiment 20 or embodiment 21, wherein step (e) is performed at a temperature in the range from 80 to 100°C.
23. A method according to any one of embodiments 20 to 22, wherein in step (e), the non-wood feedstock is passed through a screw-type conveyor.
24. A method according to any preceding embodiment, wherein the non-wood feedstock is non-wood empty fruit bunch (EFB).
25. A method for producing dissolving pulp (DP) from non-wood feedstock comprising:
- (i) pre-treating non-wood feedstock using the method as set out in any one of embodiments 1 to 24; and
 - (ii) cooking the pre-treated non-wood feedstock to produce DP.
26. A method for producing kraft pulp (KP) from non-wood feedstock comprising:
- (i) pre-treating non-wood feedstock using the method as set out in any one of embodiments 1 to 24; and
 - (ii) cooking the pre-treated non-wood feedstock to produce KP.
27. A method according to embodiment 25 or embodiment 26, wherein the non-wood feedstock is non-wood empty fruit bunch (EFB).
28. A method according to embodiment 25 or 27, comprising a further step of bleaching the DP obtained in step (ii).
29. A method according to embodiment 26 or 27, comprising a further step of bleaching the KP obtained in step (ii)
30. A method according to embodiment 28 or 29, wherein the bleaching step is an Elemental Chlorine Free (ECF) bleaching step.
31. A method according to embodiment 30, wherein the bleaching step comprises:
- (iii) treating the DP with a sequence of peroxymonosulfuric acid, chlorine dioxide, hydrogen peroxide and chlorine dioxide.
32. A method according to embodiment 28 or 29, wherein the bleaching step is a Total Chlorine Free (TCF) bleaching step.

33. A method according to any one of embodiments 28 to 32, wherein acid from the bleaching step is recycled into step (b) of the pre-treatment step (i).
- 5 34. A method according to embodiments 26, 27 or 29 to 33, wherein the KP is converted to a paper product.
35. A method according to embodiments 25, 27, 28 or 30 to 33, wherein the DP is converted to regenerated cellulose.
- 10 36. A method according to embodiment 35, wherein the regenerated cellulose is selected from the group consisting of viscose fibers, modal fibers, lyocell and MCC fibers.
37. Dissolving pulp obtainable by the method as defined in any one of embodiments 25, 27, 28 or 30 to 33.
- 15 38. Kraft pulp obtainable by the method as defined in any one of embodiments 26, 27 or 29 to 33.
- 20 39. Dissolving pulp derived from non-wood feedstock which has a silica content of less than about 80 ppm.
40. Dissolving pulp according to embodiment 37 or embodiment 39 which has a Fe content of about 10ppm or less.
- 25 41. Dissolving pulp according to any one of embodiments 37, 39 or 40, which has a Ca content of about 75 ppm or less.
42. Dissolving pulp according to any one of embodiments 37 or 39 to 41 which has an ash content of about 0.12% or less.
- 30 43. Dissolving pulp according to any one of embodiments 37 to 39 to 42, which is derived from non-wood EFB.
- 35 44. Kraft pulp derived from non-wood feedstock which has a silica content of less than about 600 ppm.

45. Kraft pulp according to embodiment 38 or 44, which has an ash content of about 0.35% or less.
- 5 46. Kraft pulp according to any one of embodiments 38, 44 or 45, which is derived from non-wood EFB.
47. Use of Dissolving Pulp (DP) as defined in embodiment 37 or embodiments 39 to 43, in the manufacture of cellulosic fibers.
- 10 48. Use of Kraft Pulp (KP) as defined in embodiment 38 or embodiments 44 to 46, in the manufacture of paper.

Claims

1. A method for pre-treating non-wood feedstock comprising:
 - (a) washing non-wood feedstock;
 - 5 (b) contacting the washed non-wood feedstock with an acid solution; and
 - (c) contacting the washed non-wood feedstock with an alkali solution;wherein the method includes an additional washing step (d) between steps (b) and (c).
2. A method according to claim 1, wherein step (b) is performed before step (c).
- 10 3. A method according to any preceding claim, wherein in step (a), the non-wood feedstock is washed using water and/or steam.
4. A method according to any preceding claim, wherein in step (b), the washed non-wood
15 feedstock is contacted with the acid solution at a temperature in the range from 80 to 100°C for a duration of 30 to 60 minutes.
5. A method according to any preceding claim, wherein in step (d), the non-wood feedstock is washed using water and/or steam.
- 20 6. A method according to any preceding claim, wherein in step (c), the washed non-wood feedstock is contacted with the alkali solution at a temperature in the range from 80 to 100°C for a duration of 30 to 60 minutes.
- 25 7. A method according to any preceding claim which further comprises:
 - (f) washing the product obtained after steps (a) to (d).
8. A method for producing dissolving pulp (DP) from non-wood feedstock comprising:
 - 30 (i) pre-treating non-wood feedstock using the method as set out in any one of claims 1 to 7; and
 - (ii) cooking the pre-treated non-wood feedstock to produce DP.
9. A method for producing kraft pulp (KP) from non-wood feedstock comprising:
 - 35 (i) pre-treating non-wood feedstock using the method as set out in any one of claims 1 to 7; and
 - (ii) cooking the pre-treated non-wood feedstock to produce KP.

10. Dissolving pulp obtainable by the method as defined in claim 8.
11. Kraft pulp obtainable by the method as defined in claim 9.
- 5 12. Dissolving pulp derived from non-wood feedstock which has a silica content of less than about 80 ppm.
- 10 13. Dissolving pulp according to claim 10 or claim 12 which has a Fe content of about 10ppm or less.
14. Dissolving pulp according to any one of claims 10, 12 or 13, which has a Ca content of about 75 ppm or less.
- 15 15. Dissolving pulp according to any one of claims 10, or 12 to 14 which has an ash content of about 0.12% or less.
16. Kraft pulp derived from non-wood feedstock which has a silica content of less than about 600 ppm.
- 20 17. Kraft pulp according to claim 11 or claim 16, which has an ash content of about 0.35% by weight or less.
18. Use of Dissolving Pulp (DP) as defined in claim 10 or claims 12 to 15, in the
- 25 manufacture of cellulosic fibers.
19. Use of Kraft Pulp (KP) as defined in claim 11 or claims 16 to 17, in the manufacture of paper.
- 30 20. A method according to any one of claims 1 to 11, dissolving pulp according to any one of claims 10 or 12 to 15, kraft pulp according to any one of claims 11, 16 or 17 or a use according to claim 18 or claim 19, wherein the non-wood feedstock is non-wood empty fruit bunch (EFB).

FIG. 1

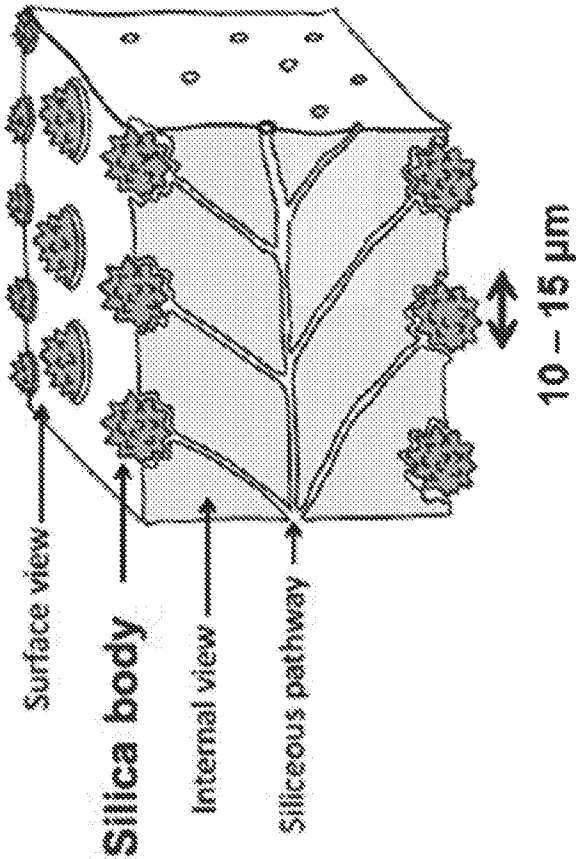


FIG. 2

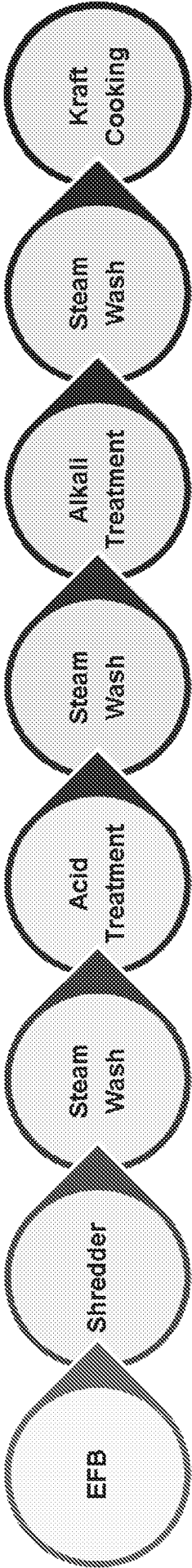


FIG. 3(a)

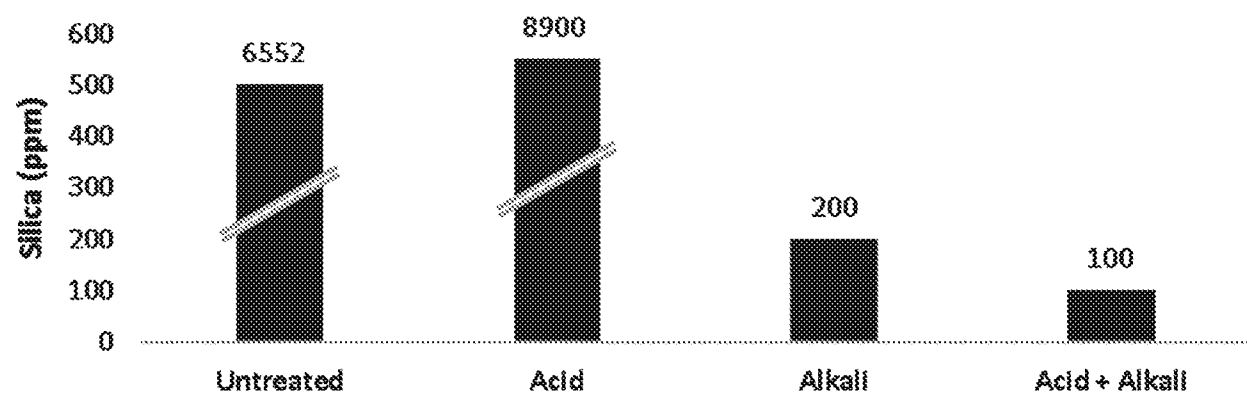


FIG. 3(b)

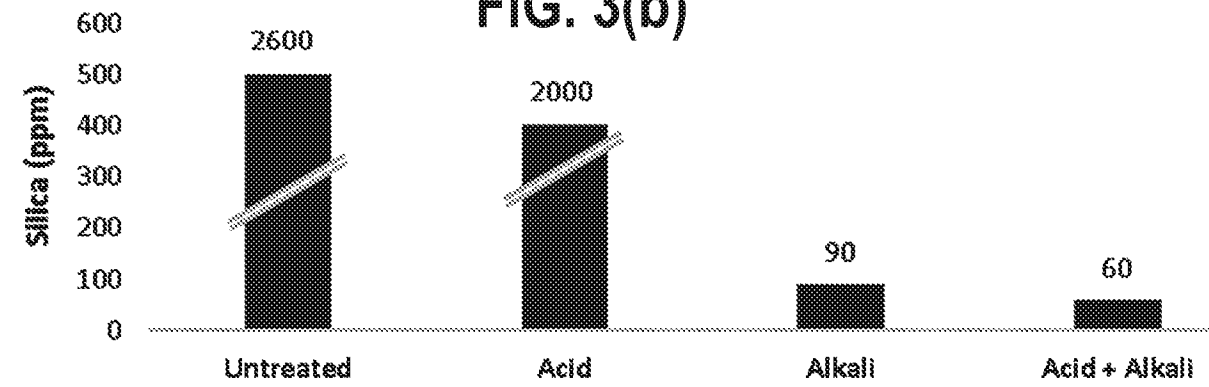


FIG. 4(a)

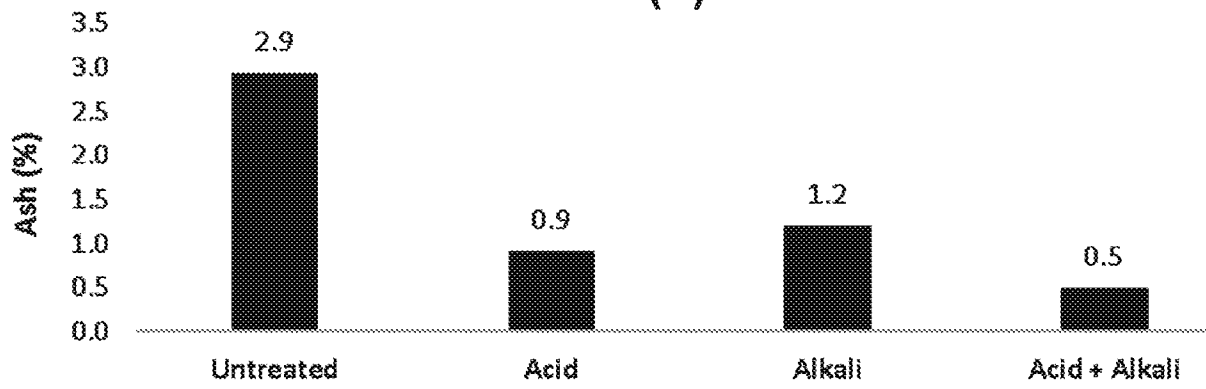


FIG. 4(b)

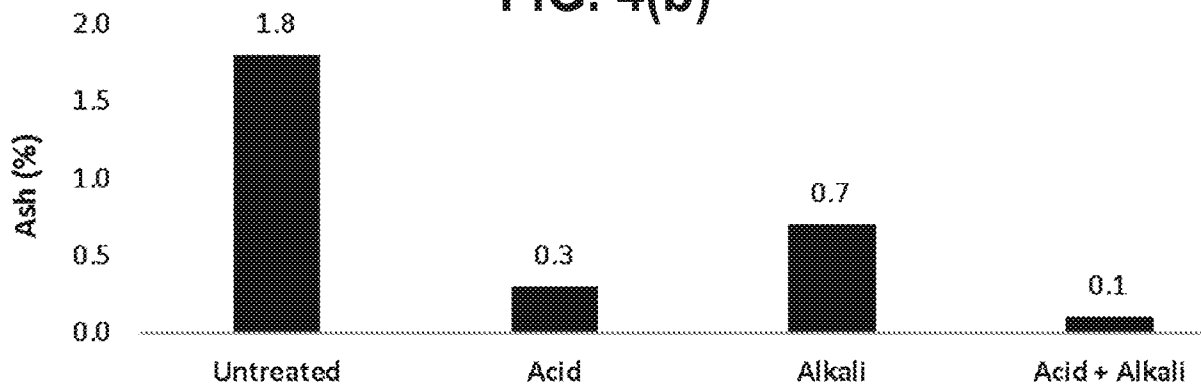
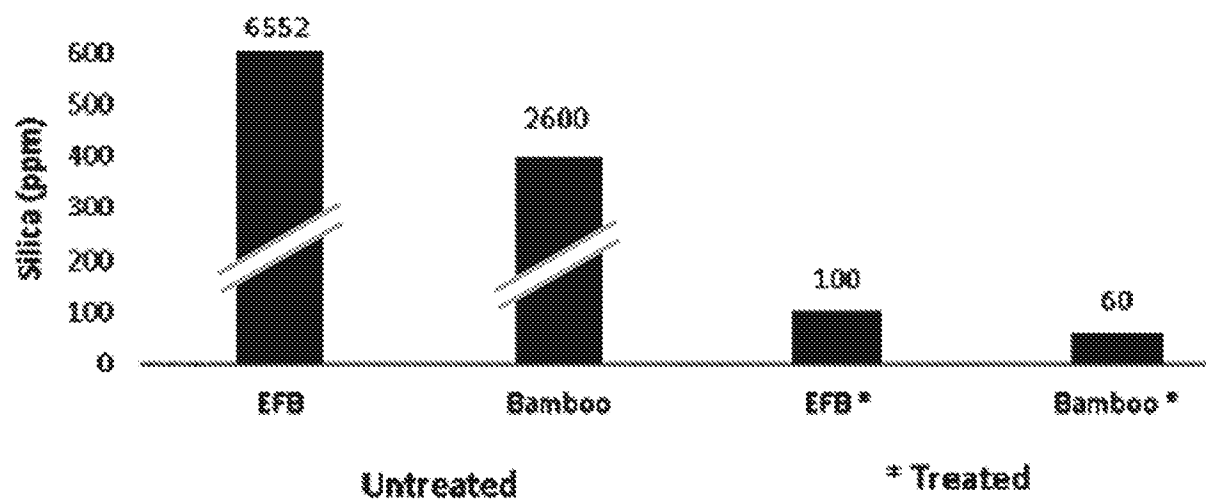


FIG. 5



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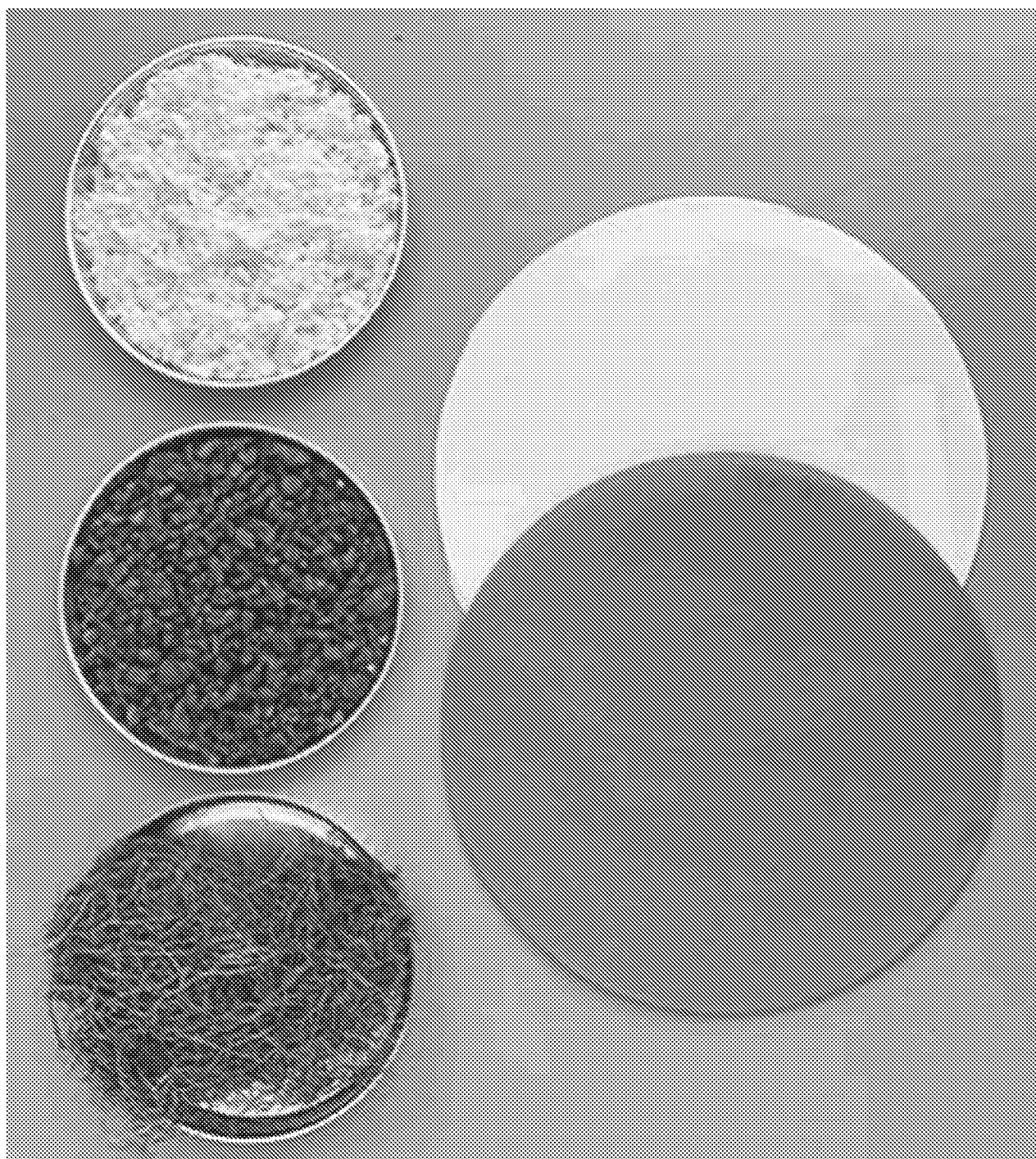
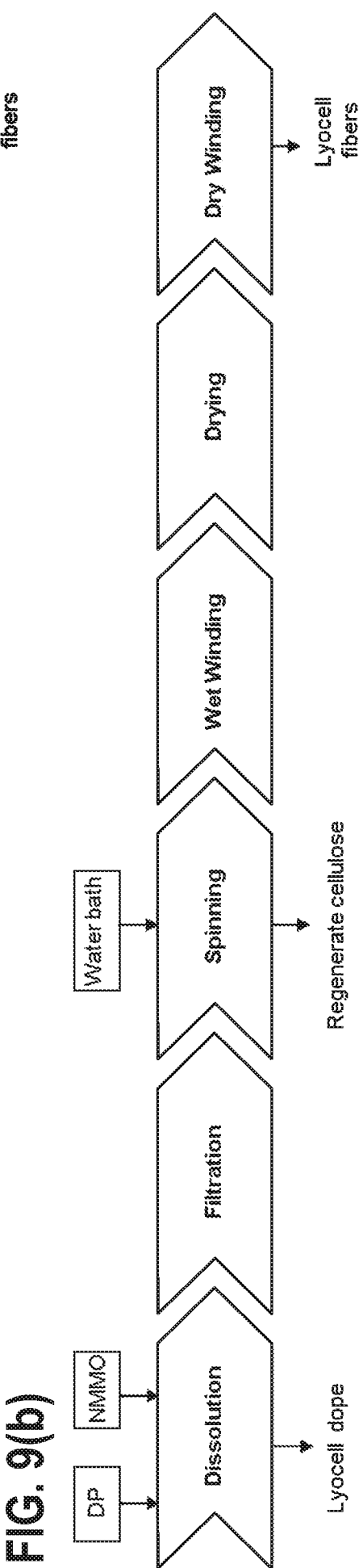
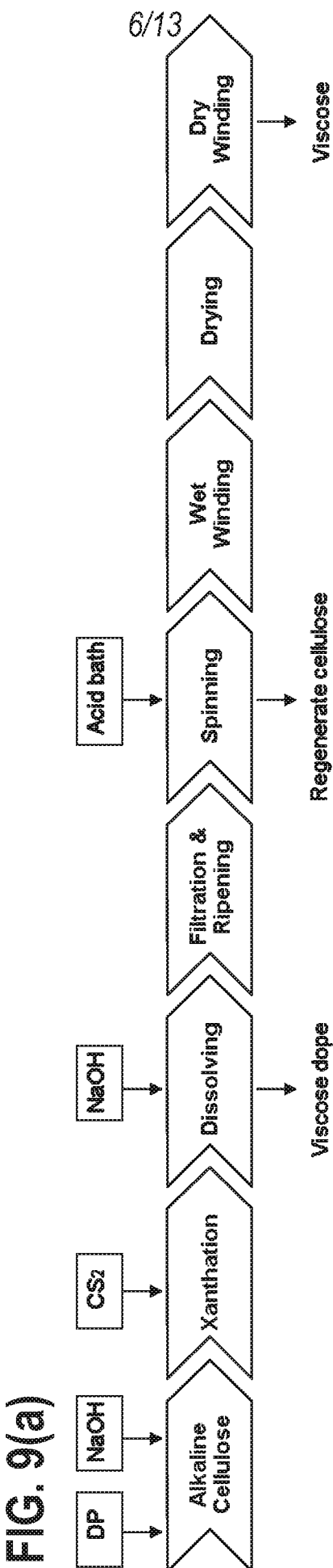
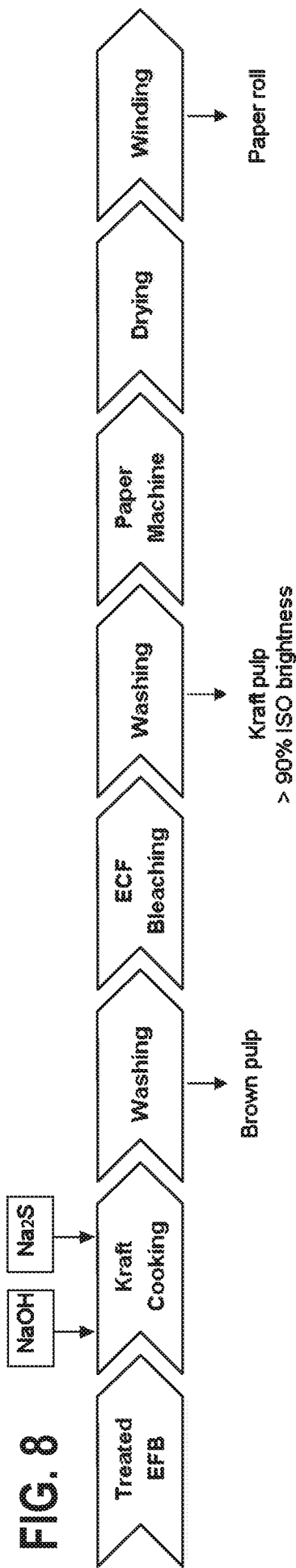


FIG. 6

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FIG. 7





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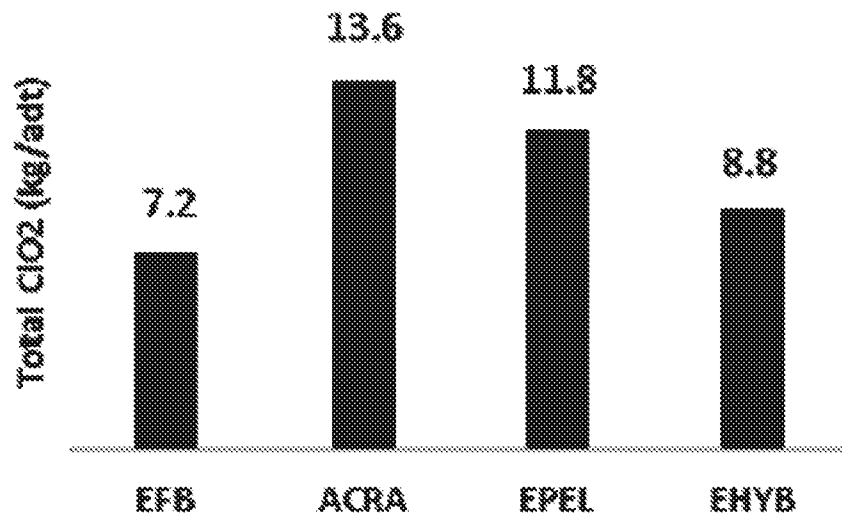
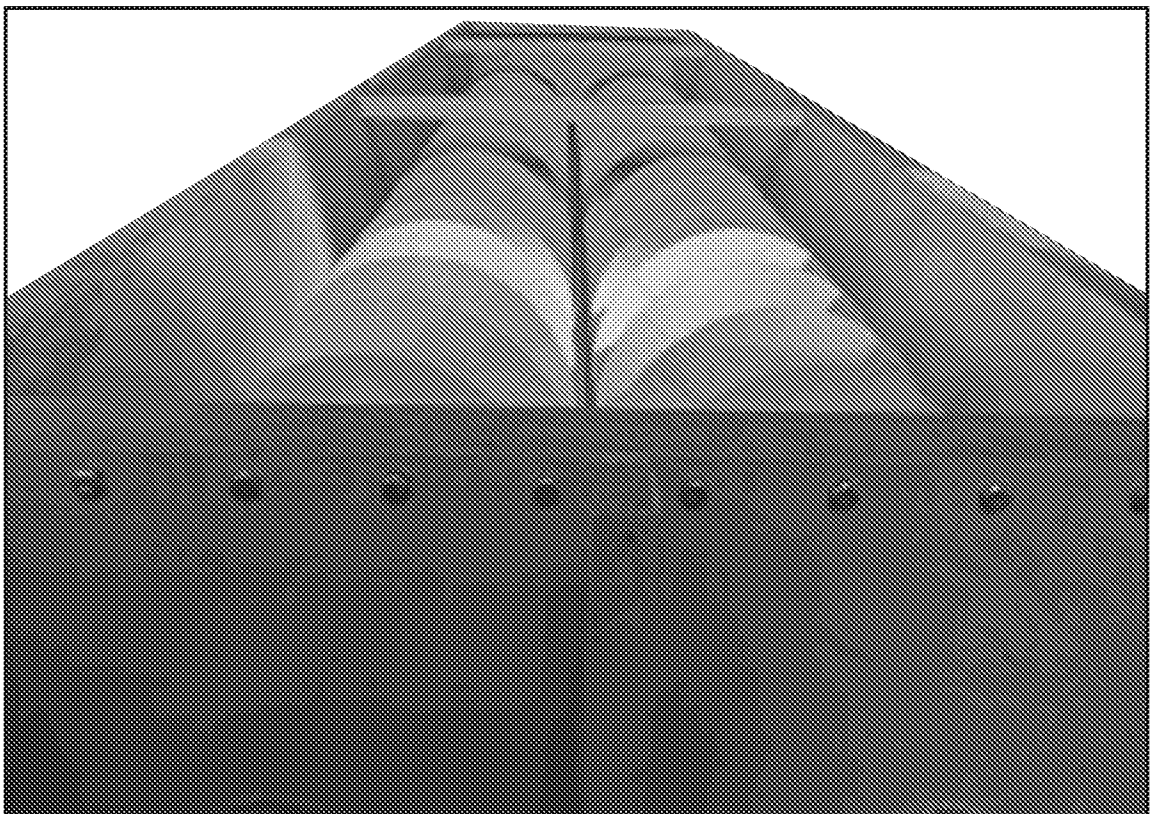
FIG. 10**FIG. 11**

FIG. 12
Mixed ratio of EFB with wood chips during mill trial

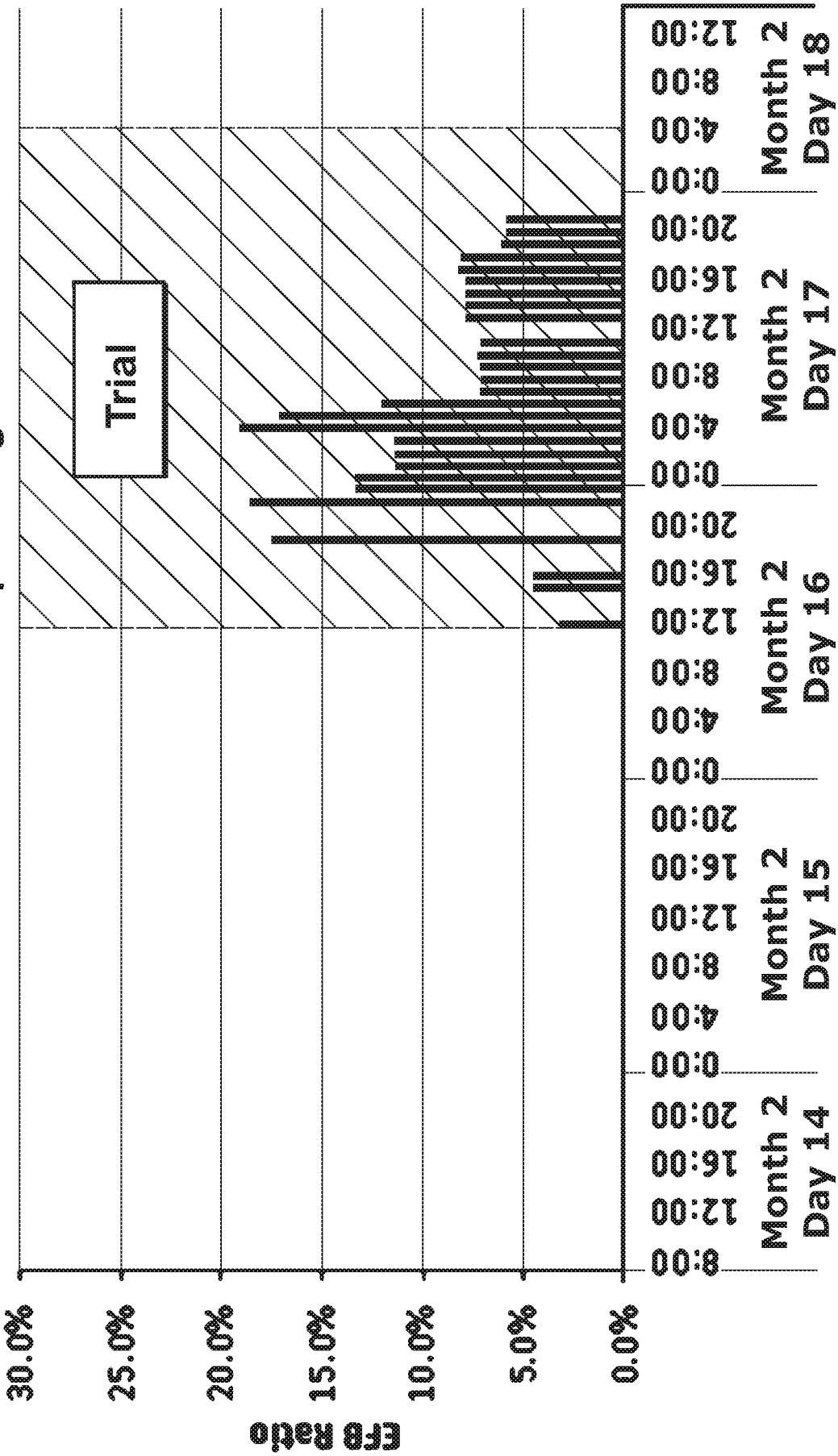


FIG. 13

Productivity (H-factor) of EFB mixed cooking with wood chips

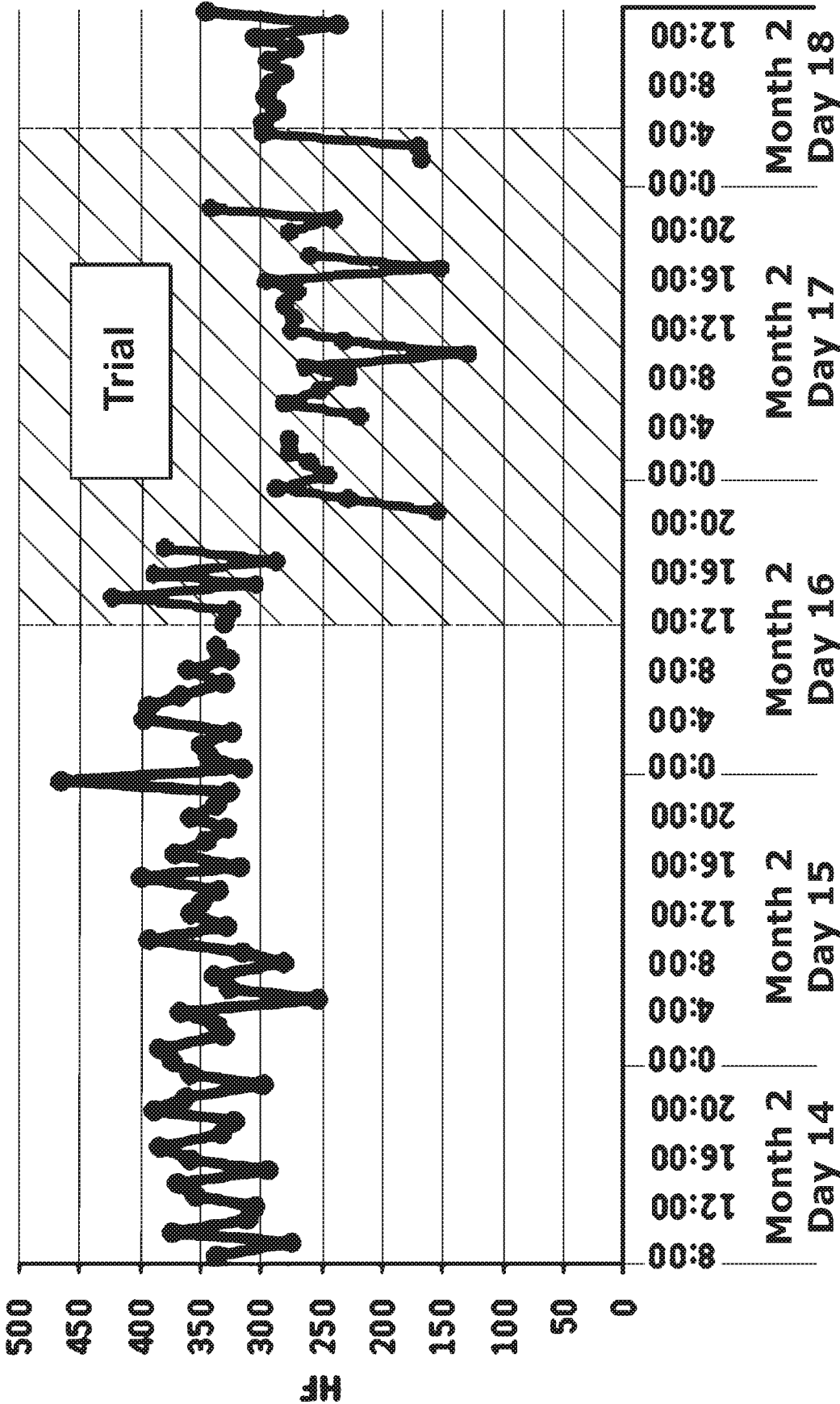


FIG. 14
Oxygen charge consumed during ODL bleaching of EFB mixed trial

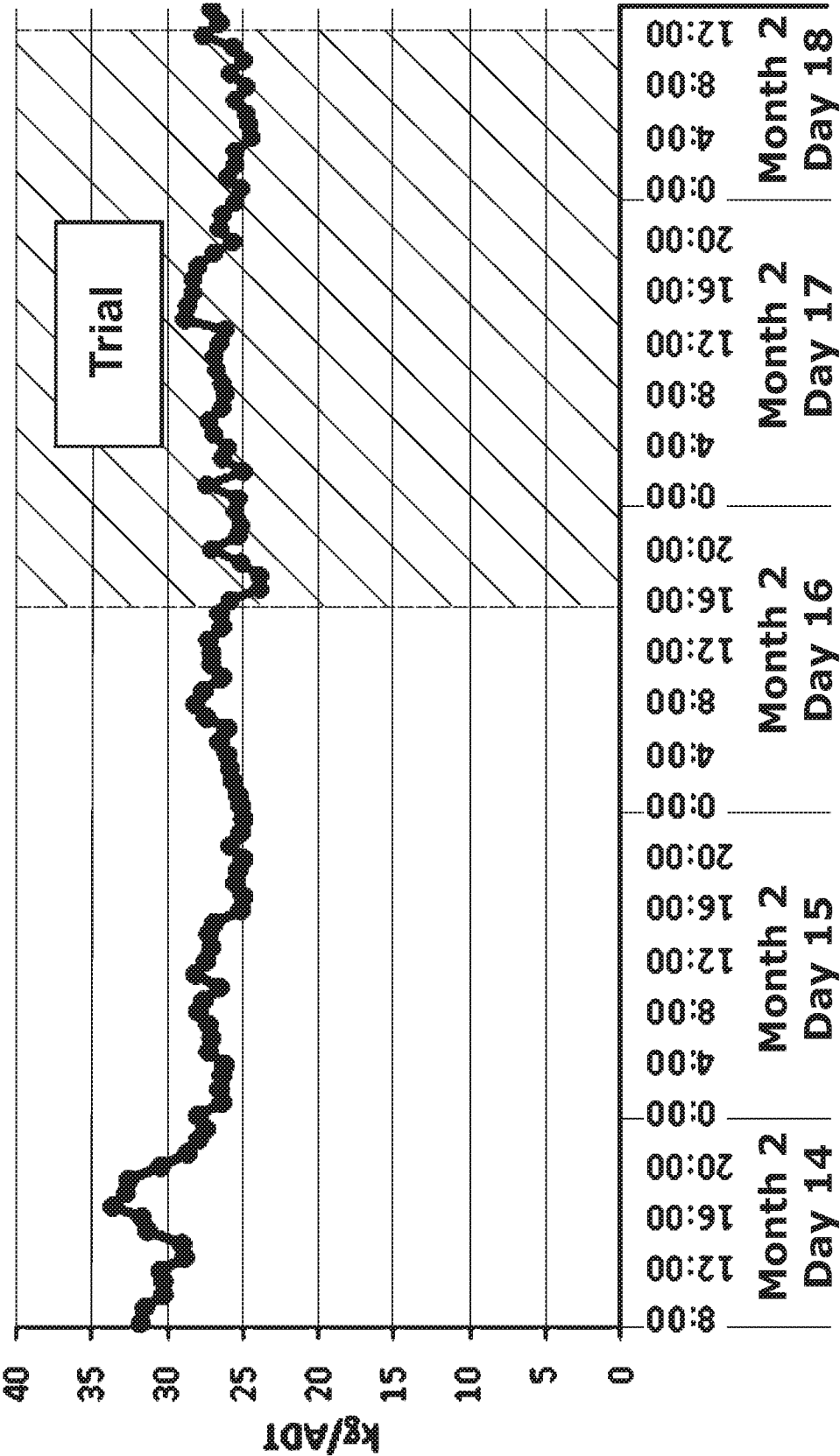


FIG. 15
Alkali charge consumed during ODL bleaching of EFB mixed trial

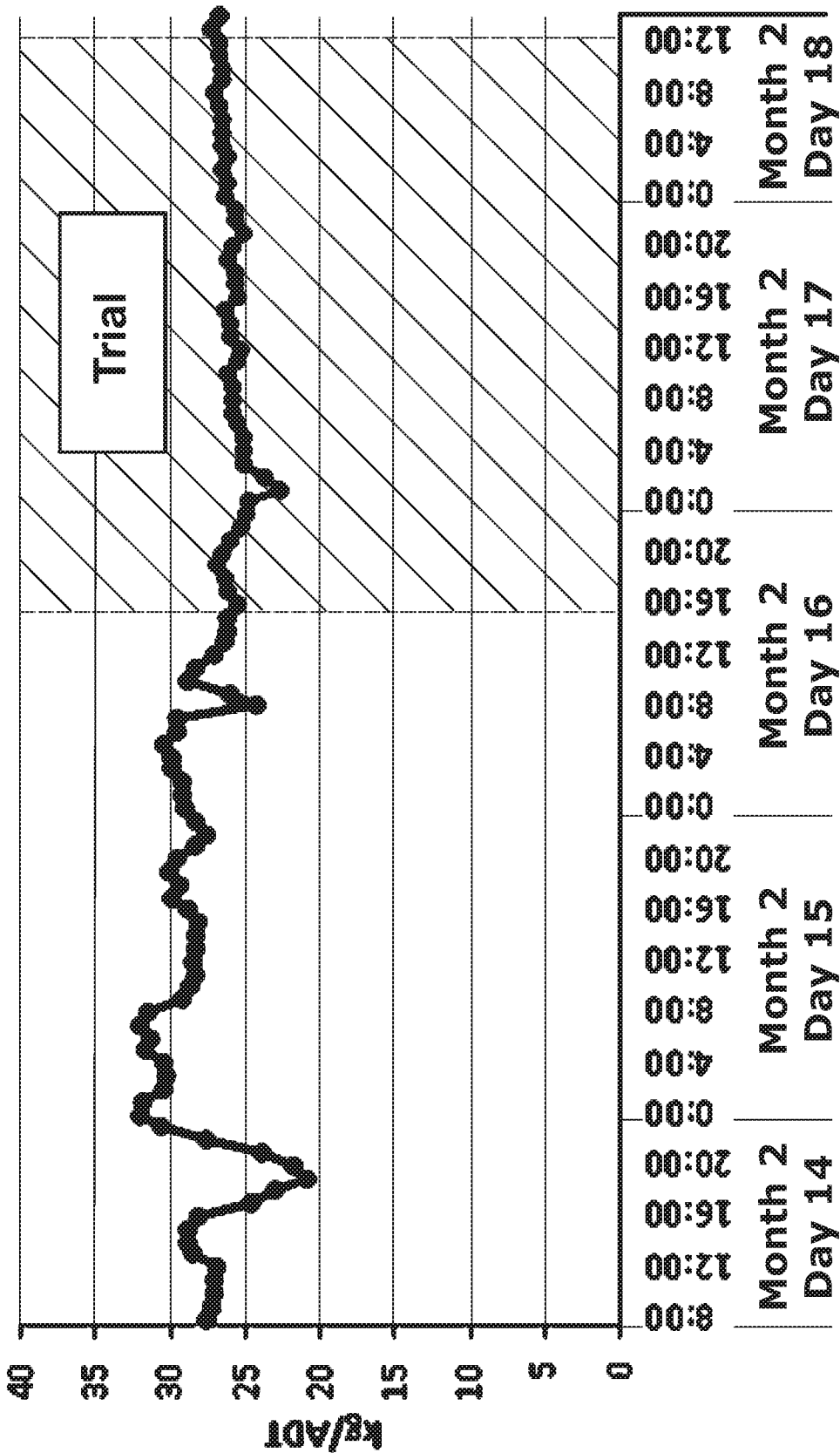


FIG. 16
Silica content of mixed EFB fully bleached kraft pulps

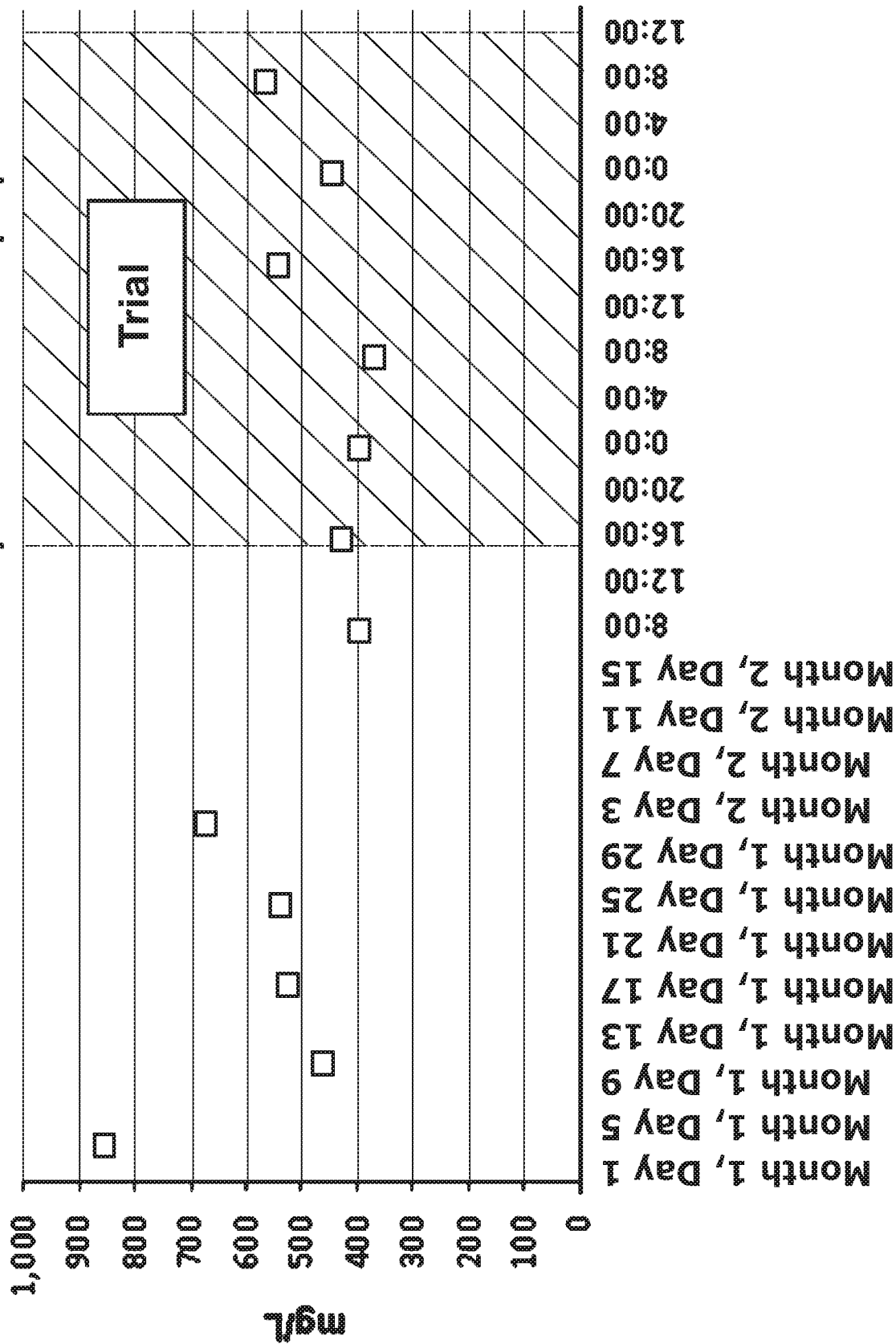
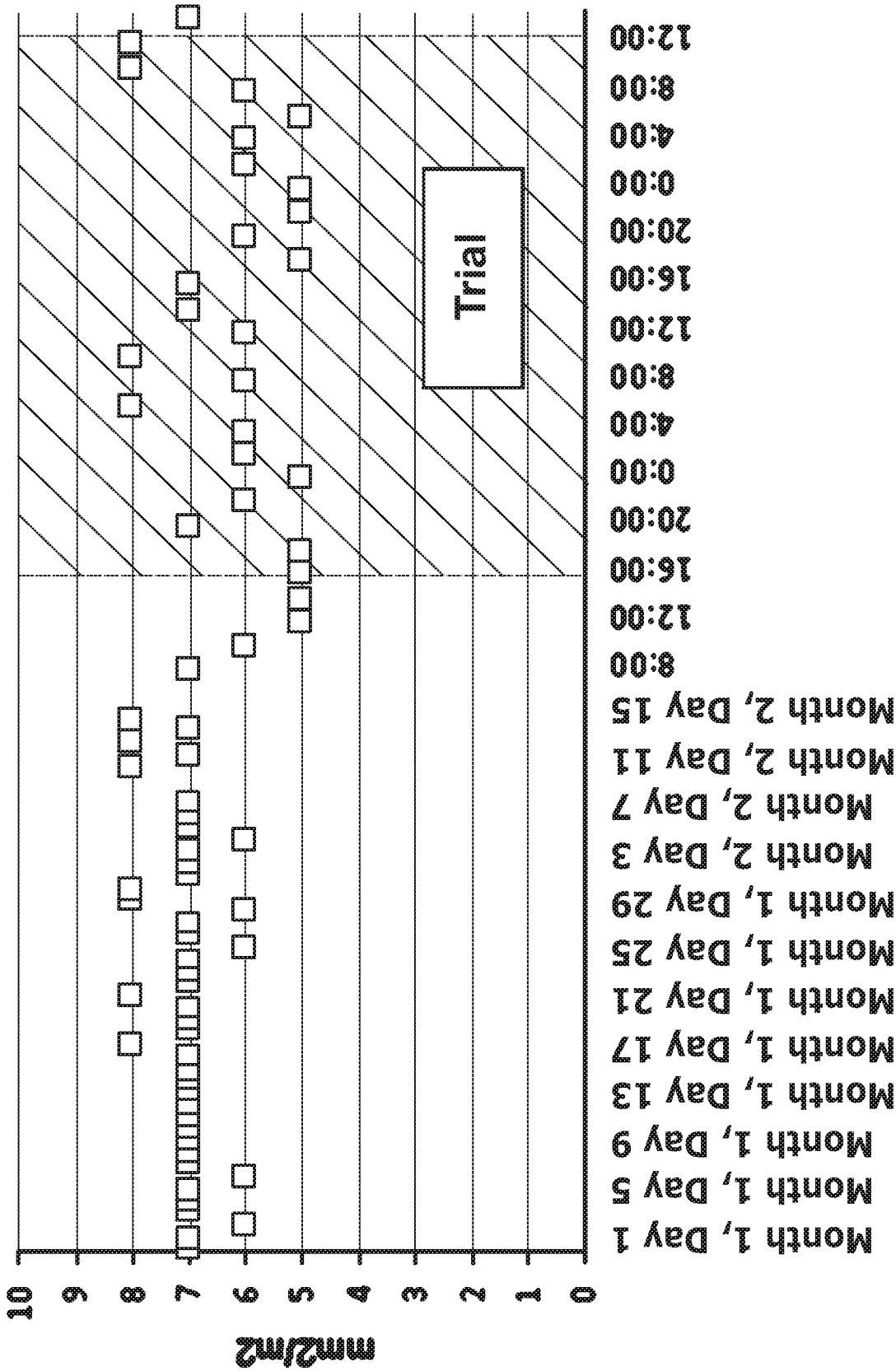


FIG. 17
Dirt spot / count of mixed EFB fully bleached kraft pulps



INTERNATIONAL SEARCH REPORT

International application No
PCT/SG2022/050268

A. CLASSIFICATION OF SUBJECT MATTER INV. D21C1/02 D21C1/04 D21C3/02 D21C3/04 D21C5/00 D21H11/12 ADD. According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) D21C D21H Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2021/006798 A1 (KIRAM AB [SE]) 14 January 2021 (2021-01-14) claims 1-16 -----	1, 12-15, 18
X	WO 2013/033405 A1 (CARGILL INC [US]; HAWKINS BRENT A [US]; MARTIN FRED [US]) 7 March 2013 (2013-03-07) claims 1-37; figure 2 -----	1-7
X	WO 01/16423 A1 (UNIV NORTH CAROLINA STATE [US]; HURTERCONSULT INC [CA]) 8 March 2001 (2001-03-08) claims 1-65; figure 1 -----	1-7
X	WO 2006/115310 A1 (LEE KWON-HYOK [KR]; WON JONG-MYOUNG [KR]) 2 November 2006 (2006-11-02) claims 1-10; figure 1 -----	1-7
-/-		
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance;; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance;; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 14 July 2022		Date of mailing of the international search report 22/07/2022
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Karlsson, Lennart

INTERNATIONAL SEARCH REPORT

International application No

PCT/SG2022/050268

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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