MULTIPLE BATCH ORGANOSOLV EXTRACTION SYSTEM

Abstract: The organosolv extraction of lignin and other materials from biomass feedstocks may be accomplished using a multi-stage batch extraction procedure such as the AL-CELL® process. For several reasons, commercial plants may benefit by processing multiple batches simultaneously through the use of multiple extractors and associated hardware. By suitably modifying the timing sequences and system design, it is possible to share or even eliminate many of the solvent accumulators typically used in such systems and thereby reduce cost and simplify system design.
This invention relates to systems and processes for batch organosolv extraction of lignin and other species from biomass feedstocks. More particularly, this invention relates to improved configurations of the extraction system.

For environmental, economic, and resource security reasons, there is an increasing desire to obtain energy and material products from bio-renewable resources and particularly from "waste" and/or non-food biomass feedstocks. The various chemical components within typical biomass can be employed in a variety of ways. In particular, the cellulose in plant matter may desirably be separated out and fermented into fuel grade alcohol. And the lignin component, which makes up a significant fraction of species such as trees and agricultural waste, has huge potential as a useful source of phenol containing chemicals for numerous industrial applications. However, most separation techniques employed by industry today are too aggressive and chemically alter the lignin component during separation to the point where it is no longer acceptable for use in most of these potential applications.

Organosolv extraction processes on the other hand are typically less aggressive and can be used to separate lignin and other useful materials from biomass without unacceptably altering or damaging the lignin. Such processes can therefore be used to maximize the value from all the components making up the biomass. Organosolv extraction processes however typically involve extraction at higher temperatures and pressures with a flammable solvent than other industrial methods and thus are generally more complex and expensive. While large scale commercial viability had been demonstrated decades ago from a technical and operation perspective, organosolv extraction has not, to date, been considered economical.

An advantageous organosolv extraction method and associated system are disclosed in US4764596. The general method described therein is known as the ALCELL® process and involves multiple batch-wise extractions. It had been used successfully in a large scale commercial operation many years ago. The method disclosed is a three stage extraction technique that initially uses liquors extracted from previous batches of biomass in two of the stages and then uses fresh solvent for the final third stage. The system shown comprises a single extractor and three solvent accumulators for storing the appropriate solvents for the three stages.

Specifically, Fig. 1 (reproduced from US4764596) depicts the important components in the organosolv extraction system. A supply of appropriate biomass (e.g. wood chips) is loaded into extractor 2. After pre-heating with low pressure steam, the biomass is contacted
with three different ethanol/water extraction solutions under conditions of elevated
temperature and pressure. The first extraction solution employed is a twice-used "primary
solvent" (from previous biomass batches) obtained from primary solvent accumulator 3.
Lignin and other materials are then extracted from the biomass into the primary solvent and
the resulting "black liquor" is displaced into recovery feed accumulator 5. The second
extraction solution is a once-used "secondary solvent" (from a previous biomass batch)
obtained from secondary solvent accumulator 6. Again, material is extracted from the
biomass into the secondary solvent and the less-concentrated resulting black liquor is
displaced into primary solvent accumulator 3. Finally, the third extraction solution is a fresh
ethanol/water mixture obtained from fresh solvent accumulator 7. Again, material is extracted
from the biomass and the resulting liquor is displaced into secondary solvent accumulator 6.

In the process of US4764596, the black liquor containing lignin and other extracted
materials still remains at high temperature and pressure in recovery feed accumulator 5. The
next step in recovering the dissolved lignin from the black liquor involves flashing the liquor
into flash tank 11.

For several reasons, it may be beneficial to process multiple batches at a time in a
commercial plant comprising multiple extractors. For instance, there are many points in the
process where useful energy and/or heat is available (e.g. during flashing or cooling steps)
but also where energy is required (e.g. during pressurization or heating steps). When multiple
batches are processed out of phase with each other, the energy/heat outputs may be more
easily matched up and used for required energy/heat inputs. For example, with multiple
batches almost always supplying concentrated liquor to recovery accumulator 5, it is possible
to almost have continuous flashing of the primary extraction liquor in flash tank 11. In turn,
this provides an almost constant supply of recoverable energy for use elsewhere in the
system. A further reason to consider a multiple batch system stems from potential equipment
limitations. At some point in plant scale-up, it may not be possible or economic to scale up
the system components for a single extractor based system when a multiple extractor system
might be used instead.

Multiple batch systems can therefore be desirable. In certain other multi-stage, multi
batch chemical extraction processes, it is in general possible to consider performing a cascade
extraction where extraction liquor/solvents are moved from one extractor to another in a
series cascade. However, in the present organosolv process, primary solvent accumulators are
generally required because the flash/filling steps lower the pressure and thus the volume of
the liquor decreases significantly. Further, because an extractor and interconnected primary
solvent accumulator are at a significantly lower pressure during the flash/fill step, they must be isolated from the rest of the accumulator network in order to prevent unwanted solvent flashing from occurring elsewhere. This generally means that a primary solvent accumulator can be fluidly connected to only one extractor at a time. Thus, a typical multiple batch organosolv extraction system may simply be configured as a parallel array of systems like those depicted in Fig. 1. Such a configuration is implied in the paper "The ALCELLTM process - A proven alternative to Kraft pulping", Pye and Lora, Tappi Journal, March 1991, Vol. 74(3) in which the illustrated system comprises multiple extractors with each expected to require its own set of accumulators but perhaps with a common recovery accumulator and a fresh solvent supply.

In order to make organosolv extraction of biomass viable commercially, simplification and/or improvements to the process and system are required.

In a multiple batch, multiple stage organosolv extraction process, we have realized that by suitably modifying the extraction timing sequences within allowable process condition requirements, it is possible to share accumulators and even eliminate accumulators for certain stages altogether in viable system configurations. The advantages of a multiple batch approach can be maintained with overall simplification of the system and a reduction in the components required and hence in capital cost.

In general, biomass processed in the organosolv extraction system undergoes a primary solvent extraction followed by a secondary solvent extraction. The solvent used for the primary solvent extraction is generally the liquor obtained from the secondary solvent extraction. The system comprises at least one, usually two or more, extractor, a number of primary accumulators, and a supply of fresh solvent. Preferably, the number of primary accumulators however is fewer than the number of extractors and at least one of the primary accumulators is fluidly connected to more than one extractor. The fresh solvent can be an aqueous mixture of ethanol and water and can be used to extract lignin and other desirable co-products from the biomass.

The process can be a two stage process (e.g. where the solvent used for the secondary solvent extraction is fresh solvent) and a fresh solvent accumulator may be incorporated for temporary storage of fresh solvent from the fresh solvent supply. For further simplicity, the fresh solvent accumulator may supply more than one extractor and thus can be fluidly connected to more than one extractor.
Alternatively, the process can be a three stage process (e.g. as in the aforementioned US4764596) in which the secondary solvent extraction is followed by a tertiary solvent extraction, and in which the solvent used for the secondary solvent extraction is the liquor obtained from the tertiary solvent extraction and the solvent used for the tertiary solvent extraction is fresh solvent. In such a system, a secondary accumulator may additionally be included for temporary storage of drain liquor from the tertiary solvent extraction. The secondary accumulator may be fluidly connected to more than one extractor.

As illustrated in the examples to follow, a preferred system can comprise six extractors wherein the first, fourth and fifth extractors are fluidly interconnected and wherein the second, third and sixth extractors are fluidly interconnected, three primary accumulators wherein the first, second, and third primary accumulators are fluidly connected to the first and second extractors, the third and fourth extractors, and the fifth and sixth extractors respectively, and a fresh solvent accumulator fluidly connected to each of the extractors and to the fresh solvent supply.

The system can comprise a recovery accumulator that is optionally fluidly connected to each of the extractors. In addition, the system can comprise a lignin recovery subsystem comprising one or more flash tanks and other conventional components. The flash tank/s is/are fluidly connected to the recovery accumulator and may be arranged in series.

With regards to operation of such a system, the process in general may comprise:

a) extracting material from the biomass in a first extractor using primary extraction solvent obtained from a primary accumulator;

b) removing the liquor from the primary solvent extraction in the first extractor;

c) extracting material from the biomass in a second extractor using primary extraction solvent obtained from the primary accumulator;

d) removing the liquor from the primary solvent extraction in the second extractor;

e) extracting material from the biomass in the first extractor using secondary extraction solvent;

f) delivering the liquor from the secondary solvent extraction in the first extractor to a primary accumulator;

g) extracting material from the biomass in the second extractor using secondary extraction solvent; and
h) delivering the liquor from the secondary solvent extraction in the second extractor to a primary accumulator.

The liquors in steps b) and d) may be delivered to a flash tank fluidly connected to the first and second extractors. Optionally, these liquors may be directed to a recovery accumulator fluidly connected between the first and second extractors and the flash tank.

In particular, with regards to operation of a preferred system comprising six extractors and three primary accumulators, the process can comprise:

a) extracting material from the biomass in first, third and fifth extractors using primary extraction solvents obtained from first, second and third primary accumulators;

b) removing the liquors from the primary solvent extractions in the first, third and fifth extractors;

c) extracting material from the biomass in second, fourth and sixth extractors using primary extraction solvents obtained from the first, second, and third primary accumulators;

d) removing the liquors from the primary solvent extractions in the second, fourth, and sixth extractors;

e) extracting material from the biomass in the first, third, and fifth extractors using secondary extraction solvent;

f) delivering the liquor from the secondary solvent extraction in the first, third, and fifth extractors to the group consisting of the first, second and third primary accumulators;

g) extracting material from the biomass in the second, fourth, and sixth extractors using secondary extraction solvent; and

h) delivering the liquor from the secondary solvent extraction in the second, fourth, and sixth extractors to the group consisting of the first, second, and third primary accumulators.

With regards to process timing, the duration of the primary solvent extraction may be an integer multiple of the duration of the secondary solvent extraction (which, in a two stage process, is the duration of the fresh solvent extraction). For instance, in some embodiments, the duration of the primary solvent extraction can be about an hour. In a two or three stage process then, the duration of the secondary and tertiary solvent extraction times can both be
about half an hour. In such a situation, the residence time for liquor in the recovery accumulator can be kept desirably brief, i.e. less than about an hour.

The following drawings illustrate certain aspects of the invention but should not be construed as limiting in any way.

Fig. 1 shows a schematic of a prior art system having a single extractor, a single primary accumulator, and a secondary accumulator and a tertiary (fresh solvent) accumulator.

Fig. 2 shows a partial schematic of an embodiment of the invention employing 6 extractors (1-6), 3 primary accumulators (11-13) and 1 tertiary (fresh solvent) accumulator (21) (only the portion showing these elements is shown).

Fig. 3 shows a partial schematic of another embodiment of the invention employing 7 extractors (1-7), 4 primary accumulators (11-14), and 1 secondary accumulator (31) (only the portion showing these elements is shown).

Fig. 4 shows a partial schematic of another embodiment of the invention employing 7 extractors (1-7), 4 primary accumulators (11-14), and 1 tertiary (fresh solvent) accumulator (21) (only the portion showing these elements is shown).

Unless explicitly defined elsewhere, the technical and scientific terms used herein have the meaning as that commonly understood by a person of ordinary skill in the relevant art. The singular terms "a", "an", and "the" include plurals unless the context clearly indicates otherwise. In a like manner, the word "or" includes "and" unless the context clearly indicates otherwise. The terms "includes" and "having" mean "comprises".

The materials, methods, apparatus, and examples described herein are intended to be illustrative and are not intended to limit the invention to only those disclosed.

In the multiple batch extraction system of the invention, at least one primary accumulator may be shared by at least two extractors. Thus, when compared to certain prior art systems, some primary accumulators can be eliminated. Further, other accumulators in the system may be shared and/or completely eliminated (e.g. secondary accumulators, recovery accumulators, tertiary accumulators where applicable). For instance, it may be possible to direct the liquor from one extraction stage and coming from one extractor, directly to another extractor to serve as solvent for an earlier extraction stage. In this way, no accumulator is needed for the earlier extraction stage.

We have realized that sharing is possible if there are a sufficient number of extractors in the system and if the duration and relative timing of the various steps in the process sequence are adjusted appropriately. In particular, sharing can be accomplished if the start and end times of key steps in the individual extraction sequence can be matched appropriately.
amongst the various extractors in the system. In order to do this, it is useful to be able to
adjust the durations of the various extraction steps such that they are the same or are related
by a simple integer multiple. In particular, the duration of the primary extraction step can
desirably be an integer multiple of the secondary and/or tertiary extraction steps. Fortunately,
from a process perspective, there is usually some flexibility with regards to acceptable
durations of the various extraction steps, especially in steps other than the primary extraction
step. The primary extraction step generally lasts as long or longer than subsequent extraction
steps and can be of the order of 1-2 hours in duration.

The multiple batch, multiple extractor system and process are suitable for the
organosolv extraction of lignin from lignocellulosic feedstocks using an aqueous ethanol
solvent mixture. Several embodiments of such systems are illustrated in the Examples below.
And more specifics for adjusting step duration and for matching the start and end of the
various steps should be easily understood by way of reference to these detailed Examples.
Those skilled in the art will readily appreciate that many other configurations and timings
may be possible within the allowable constraints for any given specific situation.

The processes provided herein comprise the displacement of one liquor with another.
For example, the primary liquor is replaced by the secondary liquor. It is preferred that one
liquor is displaced by the additional of the replacement liquor i.e. as the secondary liquor
enters the primary extractor it displaces the primary liquor. In contrast with the processes
described in US4764596, it is preferred that the displacing liquor is added from the top or the
extraction vessel and that the displaced liquor is removed from the bottom. Surprisingly, this
configuration offers advantages over the established process. For example, a higher yield of
lignin may be extracted from the biomass. Additionally, this may allow for using an extractor
for feeding a pump and therefore eliminate the need for an accumulator.

In the present disclosure we talk about liquor or other fluids being "displaced" by the
replacement liquor. However, it will be clear to the skilled person that some of the liquor to
be displaced may mix with the replacement fluids and hence a total displacement will not
occur.

It is contemplated that any embodiment discussed in this specification can be
implemented or combined with respect to any other embodiment, method, composition or
aspect of the invention, and vice versa.

Unless defined otherwise, all technical and scientific terms used herein have the same
meaning as is commonly understood by one of ordinary skill in the art to which this invention
belongs. Unless otherwise specified, all patents, applications, published applications and
other publications referred to herein are incorporated by reference in their entirety. If a
definition set forth in this section is contrary to or otherwise inconsistent with a definition set
forth in the patents, applications, published applications and other publications that are herein
incorporated by reference, the definition set forth in this section prevails over the definition
that is incorporated herein by reference. Citation of references herein is not to be construed
nor considered as an admission that such references are prior art to the present invention.

Use of examples in the specification, including examples of terms, is for illustrative
purposes only and is not intended to limit the scope and meaning of the embodiments of the
invention herein. Numeric ranges are inclusive of the numbers defining the range. In the
specification, the word "comprising" is used as an open-ended term, substantially equivalent
to the phrase "including, but not limited to," and the word "comprises" has a corresponding
meaning.

The invention includes all embodiments, modifications and variations substantially as
hereinbefore described and with reference to the examples and figures. It will be apparent to
persons skilled in the art that a number of variations and modifications can be made without
departing from the scope of the invention as defined in the claims. Examples of such
modifications include the substitution of known equivalents for any aspect of the invention in
order to achieve the same result in substantially the same way.

The present invention will be further illustrated in the following examples. However it
is to be understood that these examples are for illustrative purposes only, and should not be
used to limit the scope of the present invention in any manner.

**Example 1**

A preferred configuration for a commercial organosolv extraction system is shown in
the partial schematic of Figure 2. For simplicity, only the portion comprising the multiple
extractors and accumulators involved in solvent extraction are shown. The rest of the system
is similar to that shown in Figure 1.

The system of Figure 2 is designed for a three stage extraction of biomass and
employs 6 biomass extractors (1-6), three primary accumulators (11-13), a recovery
accumulator (not shown), no secondary accumulators, and one common tertiary fresh solvent
accumulator (21).

As shown, the top headers of the extractors are fluidly interconnected via suitable
piping and valving. The bottom headers of extractors are also fluidly interconnected together.
Here, the primary accumulators are denoted 11, 12, and 13 respectively. As shown in Figure 2, there are fluid connections between primary accumulator 11 and extractors 1, 2; between primary accumulator 12 and extractors 3, 4; and between primary accumulator 13 and extractors 5, 6. In this way, each pair of extractors shares a common primary accumulator. Appropriate plumbing is provided such that each primary accumulator can fill from each extractor in its associated pair and also such that each extractor in a pair can be filled by its associated primary accumulator.

In this Example, there is no secondary accumulator and, instead of directing liquor from the tertiary fresh solvent extraction to a secondary accumulator, the liquor is supplied directly to another extractor ready for its secondary solvent extraction.

In this Example, tertiary fresh solvent accumulator 21 is provided to temporarily hold fresh solvent for purposes of supplying fresh solvent to each of the six extractors for the tertiary fresh solvent extraction stage (i.e. accumulator 21 is shared amongst all the extractors). Appropriate fluid connections are therefore provided from fresh solvent accumulator 21 to each extractor 1, 2, 3, 4, 5, and 6.

As mentioned previously, the rest of the system (not shown) is similar to that of Figure 1. For instance, there is a sole fresh solvent supply, recovery accumulator, flash tank, lignin recovery subsystem, etc, that are commonly shared and connected appropriately to the plumbing network shown in Figure 2.

In order that there are no conflicts in operation between batches and that the individual processes/stages are carried out within acceptable processing limits, there are restrictions on the timing sequence. Table 1 shows a potentially suitable, practical timing sequence over a complete cycle for this multiple extractor system.

For an individual batch, the sequence is to first fill an extractor with biomass chips (denoted "chip fill" in Table 1). Next steam is introduced to pre-heat the biomass ("pre-steam") and is continued until sufficient oxygen is removed to reduce the risk of combustion and to permit safe operation. Because of the necessity of maintaining the correct sequence and timing for each batch in the present processes it may be convenient to introduce a hold period after the pre-steam ("hold") to account for any delays during chip-fill and pre-steam.

Then primary extraction solvent from a primary accumulator is flash filled into the extractor and heated to the target extraction temperature ("flash fill & heat-up"). In this example the duration of this step is 1 hour, there are 6 extractors undergoing this step in series (i.e. one extractor is always undergoing this step over the 6 hour period), and the overall cycle time of
the system is 6 hours, this arrangement desirably results in a leveling out of the heating requirements for the overall system.

The primary solvent extraction then is allowed to take place over about a 60 minute period. Towards the end of this period, typically 10-15 minutes before the end, some of the liquor is removed and sent to the recovery accumulator.

Table 1 shows the timing sequences for all the extractors, the primary accumulators, and the common tertiary fresh solvent accumulator in the system shown. Table 1 also shows the timing sequence for the common recovery accumulator. Here, a complete cycle takes 6 hours and each cycle is 1 hour out of phase with the next.

From a timing point of view, having the duration of the "secondary extr" phase equal the duration of the "tertiary extr" phase (1/2 hour here) and in turn having the duration of the "primary extr" phase be an integer multiple of that (2 times that or 1 hour) allows multiple sequences like this to proceed without conflicts in the system shown. The start and end times of various steps in the sequence for one batch can conveniently coincide with those of another batch (or batches) in the multiple batch system, thereby making sharing of accumulators and/or elimination of accumulators possible. Further, a desired primary extraction time of about an hour can be maintained. The secondary and tertiary extraction times are considered not as crucial and could therefore be modified accordingly. The energy and/or heat inputs and outputs within the system are spread out, thereby making it possible to better employ the latter in meeting the needs of the former.

With this arrangement, the number of primary accumulators required per extractor has been reduced in half. No secondary accumulator is needed, and a tertiary accumulator can be shared by all extractors.

**Example 2**

The partial schematic shown in Figure 3 shows an alternative embodiment for a commercial organosolv extraction system. Again, only the portion comprising the multiple extractors and accumulators involved in solvent extraction are shown. The rest of the system is similar to that shown in Figure 1.

The system in Figure 3 is also designed for a three stage extraction of biomass and employs seven biomass extractors (1-7), four primary accumulators (11-14), one secondary accumulator (31), and no tertiary fresh solvent accumulator. Like numerals as those used in Figure 2 have been employed in Figure 3. There is however seventh extractor 7, fourth primary extractor 14, secondary accumulator 31 and no tertiary fresh solvent accumulator.
As in the preceding Example, associated Table 2 shows a potentially suitable, practical timing sequence over a complete cycle for this multiple extractor system. Like in the preceding Example, here the duration of the primary extraction is also 1 hour but now 1 hour is also provided for the secondary and tertiary fresh solvent extraction steps. Also here, a complete cycle takes 7 hours with a 1 hour delay in starting up.

With this arrangement, many of the advantages of the previous Example are still generally obtained, but longer secondary and tertiary extraction times can be employed if desired. This Example permits for a slower displacement of the liquor with less mixing of the liquors which is believed to result an improved displacement process.

**Example 3**

The partial schematic shown in Figure 4 shows yet another alternative embodiment for a commercial organosolv extraction system. Again, only the portion comprising the multiple extractors and accumulators involved in solvent extraction are shown. The rest of the system is similar to that shown in Figure 1.

The system in Figure 4 is also designed for a three stage extraction of biomass and employs seven biomass extractors (1-7), four primary accumulators (11-14), no secondary accumulator, and a common tertiary fresh solvent accumulator (21). (Note: although not apparent in the Figure, a fresh solvent accumulator is generally optional and here would be relatively small). Like numerals as those used in the previous Figures have been employed in Figure 3.

Also as in the preceding Examples, associated Table 3 shows a potentially suitable, practical timing sequence over a complete cycle for this multiple extractor system. Here the duration of the various extraction stages are all 1 hour as in Example 2. However, here a complete cycle takes 7.5 hours with a 1-1.5 hour delay in starting up.

With this arrangement, the advantages of Example 2 can still be generally obtained. However, secondary accumulator 31 can be eliminated at the expense of a modest increase in overall cycle time. As will be apparent to those skilled in the art, many alterations and modifications are possible in the practice of this invention without departing from the spirit or scope thereof. For instance, the principles of the invention may be applied to a two-stage extraction process or an extraction process with three or more stages. Accordingly, the scope of the invention is to be construed in accordance with the substance defined by the following claims.
TABLE 1

Assume 6 hour cover-to-cover with a 60 minute delay from starting one batch extractor to the next extractor.

<table>
<thead>
<tr>
<th>Time (hours)</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extractor #2:</td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
<td>Primary extr</td>
<td>Secondary Extr</td>
<td>Tertiary Extr</td>
<td>Tert drain</td>
<td>Vent</td>
</tr>
<tr>
<td>Extractor #3:</td>
<td>Strip</td>
<td>Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
<td>Primary extr</td>
<td>Secondary Extr</td>
</tr>
<tr>
<td>Extractor #4:</td>
<td>Ventr</td>
<td>Strip</td>
<td>Sluice</td>
<td>Strip</td>
<td>Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
</tr>
<tr>
<td>Extractor #6:</td>
<td>FF/HU cont’d</td>
<td>Primary extr</td>
<td>Secondary Extr</td>
<td>Tertiary Extr</td>
<td>Tert drain</td>
<td>Vent</td>
<td>Strip</td>
</tr>
<tr>
<td>Primary Accum 11:</td>
<td>Fill from Ext 2</td>
<td>Flashfill/heat-up Extr 1</td>
<td>Primary Extr, Extr 1</td>
<td>Fill from Extr 3</td>
<td>Flashfill/heat-up Extr 2</td>
<td>Primary Extr, Extr 2</td>
<td></td>
</tr>
<tr>
<td>Primary Accum 12:</td>
<td>FF/HU E/ E cont’d</td>
<td>Primary Extr, Extr 6</td>
<td>Fill from Extr 6</td>
<td>Flashfill/heat-up Extr 5</td>
<td>Primary Extr, Extr 5</td>
<td>Fill from Extr 5</td>
<td>FF/HU E6</td>
</tr>
<tr>
<td>Primary Accum 13:</td>
<td>FF/HU E cont’d</td>
<td>Primary Extr, Extr 6</td>
<td>Fill from Extr 6</td>
<td>Flashfill/heat-up Extr 5</td>
<td>Primary Extr, Extr 5</td>
<td>Fill from Extr 5</td>
<td>FF/HU E6</td>
</tr>
</tbody>
</table>

Secondary Accum: Since Secondary is pumped into Extractor directly from another extractor, no accumulator is necessary.

Tertiary Accum: | Send to Extr 2 | Send to Extr 4 | Send to Extr 6 | Send to Extr 1 | Send to Extr 3 | Send to Extr 5 |
Recovery Accum: | From Extr 4 | From Extr 6 | From Extr 1 | From Extr 3 | From Extr 5 | From Extr 2 |
<table>
<thead>
<tr>
<th>Time (hours)</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Extractor #1:</strong></td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
<td>Primary extr.</td>
<td>Secondary Extr.</td>
<td>Tertiary Extr.</td>
<td>Tert drain</td>
<td>Vent</td>
<td>Strip</td>
</tr>
<tr>
<td><strong>Extractor #2:</strong></td>
<td>Strl Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
<td>Primary extr.</td>
<td>Secondary Extr.</td>
<td>Tertiary Extr.</td>
<td>Tert drain</td>
</tr>
<tr>
<td><strong>Extractor #3:</strong></td>
<td>Vent Strip</td>
<td>Sluice</td>
<td>Strl Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
<td>Primary extr.</td>
<td>Secondary Extr.</td>
</tr>
<tr>
<td><strong>Extractor #4:</strong></td>
<td>Tert cont'd</td>
<td>Tert drain</td>
<td>Vent Strip</td>
<td>Sluice</td>
<td>Strl Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
<td>Flash fill &amp; heatup</td>
</tr>
<tr>
<td><strong>Extractor #5:</strong></td>
<td>Seed cont'd</td>
<td>Tertiary Extr.</td>
<td>Tert drain</td>
<td>Vent Strip</td>
<td>Sluice</td>
<td>Strl Chip Fill</td>
<td>Pre-steam</td>
<td>Hold</td>
</tr>
<tr>
<td><strong>Extractor #6:</strong></td>
<td>Prim cont'd</td>
<td>Secondary Extr.</td>
<td>Tertiary Extr.</td>
<td>Tert drain</td>
<td>Vent Strip</td>
<td>Sluice</td>
<td>Strl Chip Fill</td>
<td>Pre-steam</td>
</tr>
<tr>
<td><strong>Extractor #7:</strong></td>
<td>Heatup cont'd</td>
<td>Primary extr.</td>
<td>Secondary Extr.</td>
<td>Tertiary Extr.</td>
<td>Tert drain</td>
<td>Vent Strip</td>
<td>Sluice</td>
<td>Strl Chip Fill</td>
</tr>
</tbody>
</table>

**Primary Accum 11:**

- Fill from E4
- Flashfill/heat-up Extr 1
- Primary Extr. Extr 1
- Fill from Extr 1
- Flashfill/heat-up Extr 5
- Primary Extr. Extr 5

**Primary Accum 12:**

- Fill from Extr 6
- Flashfill/heat-up Extr 2
- Primary Extr. Extr 2
- Fill from Extr 2
- Flashfill/heat-up Extr 6
- Primary E6

**Primary Accum 13:**

- Pri E6
- Fill from Extr 6
- Flashfill/heat-up Extr 3
- Primary Extr. Extr 3
- Fill from Extr 3
- FF/HU E7

**Primary Accum 14:**

- FF/HU E7
- Primary Extr. Extr 7
- Fill from Extr 7
- Flashfill/heat-up Extr 4
- Primary Extr. Extr 4
- Fill from E4

**Secondary Accum 31:**

- Fill from E4
- Fill from E5
- Fill from E6
- Fill from E7
- Fill from E1
- Fill from E2
- Fill from E3
- Send to E5
- Send to Extr 6
- Send to Extr 7
- Send to Extr 1
- Send to Extr 2
- Send to Extr 3
- Send to Extr 4
- Send to E5

**Tertiary Accum:**

None required since always a tertiary extraction being performed - just keep pump and HX running.

**Recovery Accum:**

- Fill from Extr 6
- Fill from Extr 1
- Fill from Extr 3
- Fill from Extr 5
- Fill from E5
- Fill from Extr 7
- Fill from Extr 2
- Fill from Extr 4
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**Primary Accum 11:**
- Flash fill/heat-up Extr 5
- Primary Extr, Extr 1
- Fill from Extr #4
- Fill from Extr #3
- Flash fill/heat-up Extr 9
- Primary Extr, Extr 5
- Fill from Extr #4

**Primary Accum 12:**
- Fill from Extr #5
- Flash fill/heat-up Extr 7
- Primary Extr, Extr 2
- Fill from Extr #2
- Flash fill/heat-up Extr 8
- Primary Extr, Extr 6
- Fill from Extr #3
- Flash fill/heat-up Extr 7

**Primary Accum 13:**
- Primary Extr, Extr 7
- Fill from Extr #6
- Flash fill/heat-up Extr 4
- Primary Extr, Extr 8

**Secondary Accum:** None required since spent tertiary liquor is drained from one extractor and directly pumped into another extractor.

**Tertiary Accum:**
- Probably need a small tertiary accumulator to fill gap in sequence, otherwise pump and heat exchanger is shutdown and restarted once every 7 hours.

**Recovery Accum:**
- Tertiary liquor is flowing
- No tertiary liquor is flowing

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Claims

1. A system for multiple batch organosolv extraction of material from a biomass feedstock, in which the biomass undergoes a primary solvent extraction followed by a secondary solvent extraction, and in which the solvent used for the primary solvent extraction is the liquor obtained from the secondary solvent extraction, comprising
   at least two extractors;
   a number of primary accumulators that is fewer than the number of extractors and wherein at least one of the primary accumulators is fluidly connected to more than one extractor; and
   a supply of fresh solvent.

2. The system of claim 1 wherein the fresh solvent comprises ethanol and water and wherein the extracted material comprises lignin.

3. The system of claim 1 comprising a fresh solvent accumulator for temporary storage of fresh solvent from the fresh solvent supply.

4. The system of claim 1 wherein the secondary solvent extraction is followed by a tertiary solvent extraction, the solvent used for the secondary solvent extraction is the liquor obtained from the tertiary solvent extraction, and the solvent used for the tertiary solvent extraction is fresh solvent.

5. The system of claim 4 comprising:
   six extractors wherein each extractor is fluidly interconnected with at least one other extractor;
   three primary accumulators wherein each primary accumulator is fluidly connected to at least two extractors; and
   a fresh solvent accumulator fluidly connected to each of the extractors and to the fresh solvent supply.

6. The system of claim 1 comprising a recovery accumulator fluidly connected to each of the extractors.
7. The system of claim 6 comprising a lignin recovery subsystem, the subsystem comprising at least one flash tank fluidly connected to the recovery accumulator.

8. The system of claim 7 comprising more than one flash tank arranged in series, in parallel, or a combination thereof.

9. A system for multiple batch organosolv extraction of material from a biomass feedstock, said system comprising at least two fluidly connected extraction vessels each vessel comprising a biomass, wherein the extraction solvent is delivered from one vessel to the other vessel via the fluid connection.

10. A process for multiple batch organosolv extraction of material from a biomass feedstock using the system of claim 1, the process comprising:
   a) extracting material from the biomass in a first extractor using primary extraction solvent obtained from at least one primary accumulator;
   b) removing the liquor from the primary solvent extraction in the first extractor;
   c) extracting material from the biomass in a second extractor using primary extraction solvent obtained from at least one primary accumulator;
   d) removing the liquor from the primary solvent extraction in the second extractor;
   e) extracting material from the biomass in the first extractor using secondary extraction solvent;
   f) delivering the liquor from the secondary solvent extraction in the first extractor to a primary accumulator;
   g) extracting material from the biomass in the second extractor using secondary extraction solvent; and
   h) delivering the liquor from the secondary solvent extraction in the second extractor to a primary accumulator.

11. The process of claim 10 wherein the fresh solvent comprises ethanol and water and wherein the material comprises lignin.

12. The process of claim 10 wherein the solvent used for the secondary solvent extraction is fresh solvent.
13. The process of claim 10 comprising a tertiary solvent extraction of the biomass wherein
the solvent used in the secondary solvent extraction is the liquor from the tertiary solvent
extraction and the solvent used in the tertiary solvent extraction is fresh solvent.

14. The process of claim 10 wherein the system comprises a fresh solvent accumulator and
the process comprises: delivering fresh solvent to the fresh solvent accumulator, and
delivering fresh solvent from the fresh solvent accumulator to the first and second
extractors.

15. The process of claim 10 wherein the liquors in steps b) and d) are delivered to a flash tank
fluidly connected to the extractors.

16. The process of claim 15 wherein the liquors in steps b) and d) are initially directed to a
recovery accumulator fluidly connected between the extractors and the flash tank.

17. The process of claim 10 wherein the duration of the primary solvent extraction is
essentially an integer multiple of the duration of the secondary solvent extraction.
FIG. 1

PRIOR ART
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

IPC: BOW 11/02 (2006.01) , C07G 1/00 (201 1.01) , C08H 6/00 (2010.01)

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)
IVO, B01D (2006.01) , C07G (201 1.01) , C08H (2010.01) , D21C (2010.01)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
EPDLOC. XFVLL. Canadian Patents Database (keywords: extraction, biomass, feedstock, ethyl alcohol, lignin, black liquor, solvent, accumulator, extractor, ALCELL, organosols )

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C. See patent family annex.

Date of the actual completion of the international search
20 April 201 1 (20-04-2011)

Date of mailing of the international search report
14 June 201 1 (14-06-2011)

Name and mailing address of the ISA/CA
Canadian Intellectual Property Office
Place du Portage 1, C114 - 1st Floor, Box PCT
50 Victoria Street
Gatineau, Quebec K1A 0C9
Facsimile No.: 001-819-953-2476

Authorized officer
Pierre Cuerrier (819) 997-4379
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