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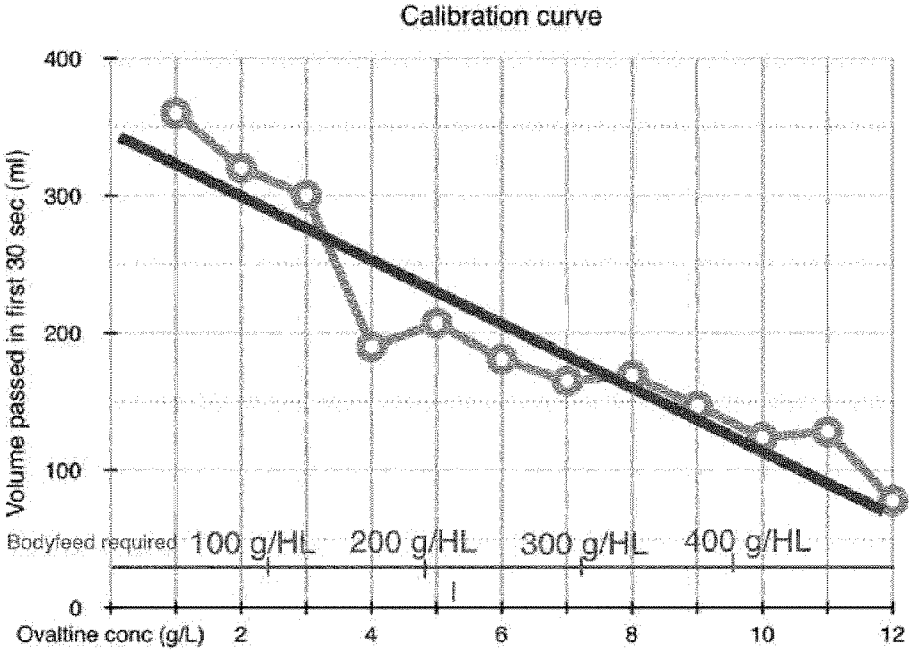


FIG. 1

(57) **Abstract:** Apparatus and methods that enable the selection of an appropriate quantity and/or grade of filter aid to filter various feed materials.

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# APPARATUS AND METHOD FOR SELECTING FILTER AID

## TECHNICAL FIELD

The present invention relates generally to apparatus and methods that enable the selection of an appropriate quantity and/or grade of filter aid to filter various feed materials.

## BACKGROUND

Particulate mineral materials such as diatomaceous earth and perlite can be used to aid filtration of various feed materials, for example feed materials in the food and beverage industry such as precursors to sugar syrups, wine, beer, oils, milk, fruit juices, water and other soft drinks and feed materials in the (non-edible) oil and biodiesel industry. The filtration may, for example, assist in removing contaminants from the feed material and/or increase the clarity of the feed material.

The appropriate type or filter aid and amount of filter aid used may vary depending on the particular process involved, the desired processing parameters and desired end product. A manufacturer may need to test a number of different types of filter aid in a number of different quantities before selecting an appropriate type and amount of filter aid. It is therefore desirable to provide alternative and/or improved methods that enable an appropriate quantity and/or grade of filter aid to be selected.

## SUMMARY

In accordance with a first aspect of the present invention there is provided method for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

a) filtering a first test sample of the feed material with a first concentration of a test filter aid;

b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

c) using a calibration curve to select the concentration of filter aid to use based on the volume of filtrate produced or time taken measured in step b).

In certain embodiments of the first aspect of the present invention, the method further comprises:

d) filtering a second test sample of the feed material with a second concentration of the test filter aid that is different to the first concentration of the test filter aid;

e) measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced;

f) determining a correction factor, F, by the following equation:

$$F = \frac{\Delta V_C}{\Delta V_T}$$

in which

$\Delta V_c$ is the difference in the volume of filtrate produced in the specified time period or difference in time taken to filter the specified volume, during filtration of a calibration sample of the material used to create the calibration curve with the first concentration and the second concentration of the test filter aid;

$\Delta V_T$ is the difference in the volume of filtrate produced in the specified time period or difference in time taken to filter the specified volume, during filtration of the first test sample of the feed material with the first concentration of filter aid and the second test sample of the feed material with the second concentration of filter aid; and

g) dividing the concentration of filter aid determined by the method of the first aspect of the present invention by the correction factor, F.

In certain embodiments of the first aspect of the present invention, step c) includes:

(i) equating the feed material with a certain concentration of a calibration material by selecting the concentration of calibration material that produces the same volume of filtrate when filtered with the first concentration of the test filter aid in the specified time period or by

selecting the concentration of calibration material that takes the same amount of time to filter a specified amount of filtrate using the first concentration of the test filter aid; and

(ii) selecting the concentration of filter aid to use by selecting a preferred concentration of filter aid that is associated with the concentration of calibration material that has been equated with the feed material.

In certain embodiments of the first aspect of the present invention, the method further comprises, after step b):

filtering the filtrate produced by step a) with the same concentration of the test filter aid,

measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced, and

calculating the difference in volume of filtrate produced or time taken for the first test sample of the feed material and the filtrate produced by step a),

wherein step c) comprises using a calibration curve to select the concentration of filter aid to use based on the calculated difference in volume of filtrate produced or time taken. This may, for example, be an alternative to calculating a correction factor, F.

Thus, in accordance with an alternative first aspect of the present invention there is provided a method for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

a) filtering a first test sample of the feed material with a first concentration of a test filter aid;

b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

c) filtering the filtrate produced by step a) with the same concentration of the test filter aid;

d) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced;

e) calculating the difference in volume of filtrate produced or time taken measured in steps b) and d),

f) using a calibration curve to select the concentration of filter aid to use based on the difference in volume of filtrate produced or time taken calculated in step e).

In certain embodiments of the alternative first aspect of the present invention, step c) includes:

(i) equating the feed material with a certain concentration of a calibration material by selecting the concentration of calibration material that has the same difference in volume of filtrate produced in the specified time period or the same difference in time taken to produce a specified volume of filtrate when the original calibration sample and the filtrate of the calibration sample are filtered with the first concentration of the test filter aid; and

(ii) selecting the concentration of filter aid to use by selecting a preferred concentration of filter aid that is associated with the concentration of calibration material that has been equated with the feed material.

In certain embodiments of the first aspect or alternative first aspect of the present invention, the calibration curve was obtained by the method of the second aspect or alternative second aspect of the present invention, including any embodiment thereof.

In accordance with a second aspect of the present invention there is provided a method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples;

iv) defining the relationship between the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii).

In certain embodiments, the method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material further comprises, after step ii):

filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid,

measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample, and

calculating the difference in volume of filtrate produced or time taken for each calibration sample of the feed material and the filtrate of each of the respective calibration samples produced by step i),

wherein step iv) comprises defining the relationship between the preferred concentration of filter aid determined in step iii) against the calculated difference in volume of filtrate produced or time taken.

Thus, in accordance with an alternative second aspect of the present invention there is provided a method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid;

iv) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample;

v) calculating the difference in volume of filtrate produced or time taken measured in steps ii) and iv);

vi) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples; and

vii) defining the relationship between the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced or time taken calculated in step v).

In certain embodiments, step iv) of the second aspect comprises creating a calibration curve by plotting the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or against the time taken to filter the specified volume of filtrate measured in step ii) for each of the calibration samples and determining the equation for the line of best fit. In certain embodiments, step vii) of the alternative second aspect comprises creating a calibration curve by plotting the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced or time taken calculated in step v) for each of the calibration samples and determining the equation for the line of best fit.

In certain embodiments of the second aspect or alternative second aspect of the present invention, the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;
B) measuring the rate of pressure rise over a specified time period for each concentration of filter aid used;
C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

In certain embodiments of the first, alternative first, second or alternative second aspect of the present invention, where the filtrate of a test feed material or calibration sample is further filtered using the same concentration of the test filter aid, a new sample of test filter aid is used. This means that the test filter aid that is used to filter the filtrate is the same type and concentration as the first concentration of a test filter aid but has not previously been used for filtration before.

In accordance with a third aspect of the present invention there is provided a method for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising:

a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
b. measuring the turbidity of the first filtrate;

c. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
d. measuring the turbidity of the second filtrate;
e. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced;
f. selecting a desired turbidity of the feed material after filtration and using the defined relationship to select the permeability of filter aid to use based on the desired turbidity of the feed material after filtration.

In accordance with a fourth aspect of the present invention there is provided a method for creating a calibration curve for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising:

a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
b. measuring the turbidity of the first filtrate;
c. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
d. measuring the turbidity of the second filtrate;
e. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced.

In certain embodiments of the third and fourth aspects of the present invention, one or more further test sample(s) of the feed material are filtered with one or more further filter aid(s) having different permeabilities and the turbidity of the filtrate(s) produced is also used to define the relationship between the permeability of filter aid and turbidity of the filtrate produced.

In accordance with a fifth aspect of the present invention there is provided a method comprising:

a) filtering a first test sample of a feed material with a first concentration of a test filter aid;
b) filtering the filtrate produced in step a) with the same concentration of the test filter aid;

c) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced from the filtration of step b);

d) calculating the difference in volume of filtrate produced in the specified time period or the difference in the time taken for the specified amount of filtrate to be produced for the first test sample of the feed material (as measured in step c)) and water when filtered using the same concentration of the test filter aid.

In certain embodiments of the fifth aspect of the present invention, the method further comprises measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced for a water test sample. Thus, in certain embodiments, the method further comprises filtering a sample of water with the same concentration of the test filter aid, and measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced. In certain embodiments, the volume of the first test sample of the feed material and the water test sample is the same.

In certain embodiments of the fifth aspect of the present invention, the water is distilled water.

In certain embodiments of the fifth aspect of the present invention, the method is for predicting the ease of filtration of the feed material. A lower value calculated in step d) indicates easier filtration of the feed material. In other words, the value calculated in step d) decreases with increasing ease of filtration.

In certain embodiments of the fifth aspect of the present invention, the method is for predicting time required for filtration of the feed material. A lower value calculated in step d) indicates a shorter filtration time. In other words, the value calculated in step d) decreases with filtration time.

In certain embodiments of the fifth aspect of the present invention, the method is for designing a process for manufacturing a product, wherein the process for manufacturing comprises filtering the feed material.

In certain embodiments of the fifth aspect of the present invention, the method is for comparing the ease of filtration of one or more different feed materials, wherein the method is performed for a test sample of each feed material. Feed materials with lower values calculated in step d) are easier to filter than feed materials having higher values calculated in step d).

Certain embodiments of any aspect of the present invention may provide one or more of the following advantages:

- reduce the time taken and amount of materials required to predict the amount and/or grade of filter aid to use for filtration of a particular feed material;
- performed on small scale laboratory equipment;
- no electrical power source required;
- accurately predicts quantity of filter aid to use to give desired results;
- accurately predicts the grade of filter aid to use to give desired results.

The details, examples and preferences provided in relation to any particulate one or more of the stated aspects of the present invention will be further described herein and apply equally to all aspects of the present invention. Any combination of the embodiments, examples and preferences described herein in all possible variations thereof is encompassed by the present invention unless otherwise indicated herein, or otherwise clearly contradicted by context.

## BRIEF DESCRIPTION OF THE FIGURES

Figure 1 shows a calibration curve for use in selecting the amount of filter aid to use for filtering a sample.

Figure 2 shows an exemplary filtration device in which A is a 48 micron steel weave soldered into a retaining ring and B is a dutch weave steel support plate.

## DETAILED DESCRIPTION

Method for selecting concentration of filter aid

There is provided herein a method for selecting the concentration of filter aid to use for the filtration of a feed material. There is further provided herein a method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material. There is also provided herein a use of the calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material.

The method for selecting the concentration of filter aid to use for filtration of a feed material may select a concentration of filter aid for filtration of the feed material that is within an optimum range for concentration of filter aid. The term “optimum” may, for example, refer to the quantity of filter aid that provides an efficient filtration, for example by maintaining a desired pressure for a desired period of time, whilst reducing or minimizing the amount of filter aid that is required. The desired period of time and desired pressure may vary depending on the requirements of the manufacturer and/or the apparatus used for the filtration.

The method for selecting the concentration of filter aid to use for filtration of a feed material can be used as a single method to select the concentration of any (multiple) filter aids for filtration of the feed material. In other words, the result of the method is appropriate for all types of filter aid, even filter aids that were not used as the test filter aids to obtain the result of the method.

The method for selecting the concentration of filter aid may comprise assessing the similarity of the feed material to a calibration material and selecting a suitable concentration of filter aid based on the degree of similarity between the feed material and the calibration material. The similarity of the feed material to calibration material may be determined by measuring a particular behaviour of the test material and comparing to the behaviour of the calibration material.

For example, the feed material may be equated to a certain variant of a characteristic of the calibration material such as concentration of calibration material. There is then a predetermined preferred concentration of filter aid that is associated with that variant of the calibration material (e.g. that concentration of the calibration material) and the

predetermined preferred concentration of filter aid is selected as the concentration of filter aid to use for filtration of the feed material.

The feed material may, for example, be equated to a certain variant of a characteristic of a calibration material by comparing the behaviour of the feed material to the behaviour of the calibration material as that characteristic of the calibration material (e.g. concentration) is varied. For example, the volume of filtrate produced in a specified time period by filtering a test sample of the feed material and one or more test sample(s) of the calibration material with a test filter aid may be compared. For example, the time taken to produce a specified volume of filtrate by filtering a test sample of the feed material and one or more test sample(s) of the calibration material with a test filter aid may be compared. The one or more test sample(s) of the calibration material may each be a different variant of the characteristic of calibration material (e.g. may each be a different concentration of the calibration material).

The persons carrying out the method for selecting the concentration of filter aid to use may or may not create the calibration curve themselves. The persons carrying out the method for selecting the concentration of filter aid to use may, for example, be provided with information to enable them to correlate the degree of similarity to the calibration material with the concentration of filter aid, for example from an external source, or may, for example, determine the correlation between the degree of similarity and the concentration of filter aid themselves. For example, the persons carrying out the method for selecting the concentration of filter aid to use maybe provided with the predetermined preferred concentrations of filter aid, for example from an external source, or may, for example, determine the preferred concentrations of filter aid themselves.

The method for selecting the concentration of filter aid to use for the filtration of a feed material may comprise:

a) filtering a first test sample of the feed material with a first concentration of a test filter aid;
b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

c) using a calibration curve to select the concentration of filter aid to use based on the volume of filtrate produced or time taken measured in step b).

The level and nature of the suspended solids in the feed material may impact the control of the flow of liquid through a filter aid. It has surprisingly been found that the concentration of filter aid to use for the filtration of a feed material may be adjusted by comparing the difference in filtration of the original feed material and the filtrate of the original feed material in which the suspended solids in the original feed material have been substantially removed. This may be referred to as the particle-imposed filtration flow loss (PIFFL) and may involve calculating the difference in volume of filtrate produced in a specified time period or time taken to filter a specified volume of filtrate for the original feed material and the filtrate of the original feed material.

Therefore, the method for selecting the concentration of filter aid to use for the filtration of a feed material may comprise:

a) filtering a first test sample of the feed material with a first concentration of a test filter aid;

b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

c) filtering the filtrate produced by step a) with the same concentration of the test filter aid;

d) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced;

e) calculating the difference in volume of filtrate produced or time taken measured in steps b) and d),

f) using a calibration curve to select the concentration of filter aid to use based on the difference in volume of filtrate produced or time taken calculated in step e).

The filtrate of the test feed material may be further filtered using a new sample of the same concentration of the test filter aid. This means that the test filter aid that is used to filter the filtrate is of the same type and concentration as the first concentration of a test filter aid but has not previously been used for filtration before.

The persons carrying out the method for selecting the concentration of filter aid to use may, for example, be provided with the calibration curve, for example from an external source, or may, for example, create the calibration curve themselves.

The feed material may, for example, be any liquid that needs to be filtered. The feed material may, for example, be an aqueous or non-aqueous liquid.

The feed material may, for example, be any precursor to a non-edible oil or biodiesel. For example, the feed material may be a petroleum-based crude oil or derived from a petroleum-based crude oil. For example, the feed material may be a precursor to diesel.

Biodiesel is a form of purified alkyl esters of fatty acids generally referred to as fatty acid alkyl esters (FAAEs). Production of these FAAEs is achieved by the transesterification of animal and/or vegetable fats and/or oils or by the esterification of fatty acids, including free fatty acids (FFAs) found in degraded fat oil. Biodiesel can also be derived from triacylglycerides (also called triglycerides), which may be obtained from plant sources and/or animal fat sources. However, simply performing the esterification and/or transesterification of fatty acids is not enough to produce a usable biodiesel fuel. FFAEs contain impurities that can crystallize, foul engines, and cause numerous problems for the user. Crude biodiesel therefore may undergo filtration to obtain biodiesel that can be marketed commercially. The feed material may, for example, be crude biodiesel or any biodiesel precursor. The feed material may, for example, comprise, consist essentially of or consist of FFAEs.

The feed material may, for example, be any precursor to a food or beverage product. For example, the feed material may be a precursor of sugar syrups, beer, wine, liquor, milk, fruit juice, water or (edible) oils (e.g. animal oils or vegetable oils such as olive oil). For example, the feed material may be a precursor of beer or wine.

The first concentration of the test filter aid may, for example, range from about 100 g/HL to about 600 g/HL (HL = hectolitre = hl = hL). For example, the first concentration of the test filter aid may be equal to or greater than about 200 g/HL or equal to or greater than about 250 g/HL or equal to or greater than about 300 g/HL or equal to or greater than about 350 g/HL. For example, the first concentration of the test filter aid may be equal to or less than about 500 g/HL or equal to or less than about 450 g/HL.

The first concentration of the test filter aid may, for example, range from about 150 g/HL to about 550 g/HL, for example from about 200 g/HL to about 500 g/HL, for example from about 250 g/HL to about 450 g/HL, for example from about 300 g/HL to about 450 g/HL, for example from about 350 g/HL to about 450 g/HL. The first concentration of the test filter aid may, for example, range from about 360 g/HL to about 440 g/HL, for example from about 370 g/HL to about 430 g/HL, for example from about 380 g/HL to about 420 g/HL, for example from about 390 g/HL to about 410 g/HL, for example from about 395 g/HL to about 405 g/HL. For example, the first concentration of the test filter aid may be about 400 g/HL.

The test filter aid may, for example, be a mineral filter aid. The mineral filter aid may, for example, be diatomaceous earth, perlite or a combination thereof.

The test filter aid may, for example, be a low permeability filter aid. For example, the test filter aid may have a permeability ranging from 0 darcy to about 2 darcy. For example, the filter aid may have a permeability ranging from about 0.05 darcy to about 1.9 darcy, for example ranging from about 0.1 darcy to about 1.8 darcy, for example from about 0.15 darcy to about 1.7 darcy, for example from about 0.2 darcy to about 1.6 darcy, for example from about 0.25 darcy to about 1.5 darcy, for example from about 0.3 darcy to about 1.4 darcy, for example from about 0.35 darcy to about 1.3 darcy, for example from about 0.4 darcy to about 1.2 darcy, for example from about 0.45 darcy to about 1.1 darcy, for example from about 0.5 darcy to about 1.0 darcy. For example, the test filter aid may have a permeability ranging from about 0.01 darcy to about 1 darcy, for example from about 0.01 darcy to about 0.8 darcy, for example from about 0.01 darcy to about 0.6 darcy, for example from about 0.01 darcy to about 0.5 darcy, for example from about 0.05 darcy to about 0.4 darcy, for example from about 0.1 darcy to about 0.3 darcy, for example from about 0.1 darcy to about 0.2 darcy.

In certain embodiments, the test filter aid is diatomaceous earth having a permeability ranging from about 0 darcy to about 1 darcy, for example from about 0.01 darcy to about 0.8 darcy, for example from about 0.01 darcy to about 0.6 darcy, for example from about 0.01 darcy to about 0.5 darcy, for example from about 0.05 darcy to about 0.4 darcy, for example from about 0.1 darcy to about 0.3 darcy, for example from about 0.1 darcy to about 0.2 darcy.

In certain embodiments, the test filter aid is perlite having a permeability ranging from about 1 darcy to about 2 darcy, for example from about 1.1 darcy to about 1.95 darcy, for example from about 1.2 darcy to about 1.9 darcy, for example from about 1.3 darcy to about 1.85 darcy, for example from about 1.4 darcy to about 1.8 darcy. In certain embodiments, the test filter aid is perlite having a permeability ranging from about 1.5 darcy to about 2 darcy or from about 1.6 darcy to about 1.9 darcy or from about 1.8 darcy to about 1.9 darcy.

Permeability may, for example, be measured by the water permeability method described by Darcy.

The specified period of time for measuring the volume of filtrate produced may be equal to or less than about 10 minutes. For example, the specified period of time for measuring the volume of filtrate produced may be equal to or less than about 9 minutes or equal to or less than about 8 minutes or equal to or less than about 7 minutes or equal to or less than about 6 minutes or equal to or less than about 5 minutes. The specified time period for measuring the volume of filtrate produced may be equal to or less than about 60 seconds. For example, the specified time period for measuring the volume of filtrate produced may be equal to or less than about 55 seconds or equal to or less than about 50 seconds or equal to or less than about 45 seconds or equal to or less than about 40 seconds or equal to or less than about 35 seconds or equal to or less than about 30 seconds or equal to or less than about 25 seconds or equal to or less than about 20 seconds or equal to or less than about 15 seconds. For example, the specified time period for measuring the volume of filtrate produced may be equal to or greater than about 5 seconds or equal to or greater than about 10 seconds or equal to or greater than about 15 seconds. The specified period of time for measuring the volume of filtrate produced may, for example, be from about 5 seconds to about 60 seconds, for example from about 10 seconds to about 40 seconds, for example from about 15 seconds to about 30 seconds. The specified period of time for measuring the volume of filtrate produced may, for example, be about 30 seconds.

The specified volume of filtrate may, for example, be equal to or less than about 1000 mL. For example, the specified volume of filtrate may be equal to or less than about 900 mL or equal to or less than about 800 mL or equal to or less than about 700 mL or equal to or less than about 600 mL or equal to or less than about 500 mL. The specified volume of filtrate may, for example, be equal to or greater than about 50 mL,

for example equal to or greater than about 100 mL or equal to or greater than about 150 mL or equal to or greater than about 200 mL or equal to or greater than about 250 mL. The specified volume of filtrate may, for example, be from about 50 mL to about 1000 mL, for example from about 100 mL to about 500 mL, for example from about 50 mL to about 250 mL.

The filtering of the test samples of the feed material with test filter aid may, for example, occur at a pressure equal to or less than about 1 bar. For example, the filtering of the test samples of the feed material with test filter aid may occur at a pressure equal to or less than about 0.9 bar or equal to or less than about 0.8 bar or equal to or less than about 0.7 bar or equal to or less than about 0.6 bar or equal to or less than about 0.5 bar or equal to or less than about 0.4 bar or equal to or less than about 0.3 bar or equal to or less than about 0.2 bar or equal to or less than about 0.1 bar. The filtering of the test samples of the feed material with test filter aid may, for example, occur at a pressure ranging from about 0.01 bar to about 1 bar, for example from about 0.05 bar to about 0.5 bar, for example from about 0.05 bar to about 0.3 bar, for example from about 0.05 bar to about 0.2 bar.

There is further provided herein a method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material. The term “calibration curve” relates to a mathematical relationship between a particular characteristic of a calibration material and a preferred concentration of filter aid to use for filtration of the calibration material. Creation of the calibration curve may therefore comprise defining the relationship between a particular characteristic of a calibration material and a preferred concentration of filter aid to use for filtration of the calibration material. This may or may not involve plotting a graph. The calibration curve may therefore be expressed as a mathematical formula or may be displayed by plotting on a graph.

A feed material can then be equated to a certain variant of the characteristic of the calibration material and the preferred concentration of filter aid that is associated with that variant is selected as the concentration of filter aid to use for filtration of the feed material. The calibration curve may be created prior to performing the method for selecting the concentration of filter aid to use for the filtration of a feed material. The calibration curve may, for example, be created and then distributed to another party

and the other party may perform the method for selecting the concentration of filter aid to use for the filtration of a feed material.

The calibration curve may, for example, be created by assessing how the preferred concentration of filter aid for filtration of a calibration material changes with a particular characteristic of the calibration material. For example, it may be determined how the preferred concentration of filter aid varies with concentration of the calibration material.

The particular characteristic of the calibration material is an indicator of similarity of the feed material to the calibration material. The similarity of the feed material to the calibration material may then be assessed by comparing the feed material to variants of the particular characteristic of the calibration material (e.g. by comparing the feed material to different concentrations of the calibration material). For example, the behaviour of the feed material may be compared to the behaviour of different variants of the particular characteristic of the calibration material. For example, the volume of filtrate produced in a specified time period by filtering a test sample of the feed material and one or more test sample(s) of the calibration material with a test filter aid may be compared. For example, the time taken for a specified volume of filtrate to be produced by filtering a test sample of the feed material and one or more test sample(s) of the calibration material with a test filter aid may be compared. The one or more test sample(s) of the calibration material may each be a different variant of the characteristic of calibration material (e.g. may each be a different concentration of the calibration material).

The calibration curve may define the preferred concentration of filter aid as a function of the particular characteristic of the calibration material specified above such as concentration of the calibration material. Additionally or alternatively the calibration curve may define the preferred concentration of filter aid as a function of a particular behaviour of the calibration material that is proportional to the particular characteristic of the calibration material specified above, for example the calibration curve may define the preferred concentration of filter aid as a function of volume of filtrate produced when filtering with a test filter aid for a specified period of time or as a function of time taken for a specified volume of filtrate to be produced.

The calibration curve may, for example, be made by a method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified volume of filtrate to be produced for each calibration sample;

iii) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples;

iv) defining the relationship between the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii).

In certain embodiments, step iv) comprises creating a calibration curve by plotting the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii) for each of the calibration samples and determining the equation for the line of best fit.

The calibration curve may, for example, be made by a method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid;

iv) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample;

v) calculating the difference in volume of filtrate produced or time taken measured in steps ii) and iv);

vi) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples; and

vii) defining the relationship between the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced or time taken calculated in step v).

In certain embodiments, step vii) comprises creating a calibration curve by plotting the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced in the specified time period or the difference in time taken to filter the specified volume of filtrate calculated in step v) for each of the calibration samples and determining the equation for the line of best fit.

The calibration material may, for example, be any material that is similar in nature to the feed material. The calibration material may, for example, be any material that behaves in a similar way to the feed material. The calibration material may, for example, be equally or more difficult to filter than the feed material that is to be filtered.

The calibration material may, for example, comprise organic solids, for example organic solids in suspension. The organic solids may, for example, be compressible. The organic solids may, for example, be small, for example, have an average particle size equal to or less than about 10 μm or equal to or less than about 8 μm or equal to or less than about 6 μm or equal to or less than about 5 μm or equal to or less than about 4 μm or equal to or less than about 2μm. The calibration material may, for example, comprise one or more of malt extract (e.g. barley malt extract), milk serum, cocoa powder, sugar, oil (e.g. rapeseed oil) and/or whey. For example, the organic solids may comprise barley malt extract. In certain embodiments, the calibration material is an Ovaltine® product (e.g. original or chocolate Ovaltine®) obtained from any country, for example obtained from the UK.

The plurality of calibration samples that are used to create the calibration curve may each have a different concentration of calibration material. The concentration of calibration material in each calibration sample may, for example, range from about 0.01 g/L to about 10 g/L. For example, the concentration of calibration material in each calibration sample may range from about 0.1 g/L to about 10 g/L or from about 0.5 g/L to about 9 g/L or from about 1 g/L to about 8 g/L or from about 1.5 g/L to about 7 g/L or from about 2 g/L to about 6 g/L or from about 2.5 g/L to about 5 g/L. The concentration of calibration material in each calibration sample may, for example, range from about 0.01 g/L to about 2 g/L or from about 0.1 g/L to about 2 g/L. The difference in

concentration between the calibration sample with the highest concentration of calibration material and the calibration sample with the lowest concentration of calibration material may, for example, be at least about 1 g/L. For example, the difference in concentration between the calibration sample with the highest concentration of calibration material and the calibration sample with the lowest concentration of calibration material may be at least about 2 g/L or at least about 3 g/L or at least about 4 g/L or at least about 5 g/L. The difference in concentration between the calibration sample with the highest concentration of calibration material and the calibration sample with the lowest concentration of calibration material may, for example, be up to about 20 g/L, for example up to about 15 g/L, for example up to about 12 g/L, for example up to about 10 g/L.

The test filter aid that is used to filter the plurality of calibration samples is the same test filter aid that is used to filter the first test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above. The concentration of the test filter aid that is used to filter the plurality of calibration samples is also the same as the concentration of test filter aid that is used to filter the first test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above.

The specified time period for measuring volume of filtrate produced using the calibration samples is substantially the same as the specified time period for measuring volume of filtrate produced using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above. For example, the specified time period for measuring volume of filtrate produced using the calibration samples may be within about 5 seconds (+ or – 5 seconds) or within about 4 seconds or within about 3 seconds or within about 2 seconds or within about 1 second of the specified time period for measuring volume of filtrate produced using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above. For example, the specified time period for measuring volume of filtrate produced using the calibration samples may be the same as the specified time period for measuring volume of filtrate produced using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above.

The specified amount of filtrate produced using the calibration samples is substantially the same as the specified amount of filtrate produced using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above. For example, the specified amount of filtrate produced may be within about 2 mL (+ or – 2 mL) or within about 1.5 mL or within about 1 mL or within about 0.5 mL of the specified amount of filtrate produced when using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above. For example, the specified amount of filtrate produced using the calibration samples may be the same as the specified amount of filtrate produced when using the test sample of the feed material during the method for selecting the concentration of filter aid to use for filtration as described above.

The preferred concentration of filter aid to use for each calibration sample may be determined by filtering each calibration sample with one or more concentrations of filter aid and selecting a concentration of filter aid that gives a desired result. Preferably, the minimum concentration of filter aid that gives a desired result is selected so no excess filter aid is used unnecessarily. The desired result may vary depending on the manufacturer's requirements and the desired product.

In certain embodiments, the desired result is for a certain amount of feed material to be filtered in a certain amount of time. In other words, in certain embodiments, the desired result is to achieve a certain rate of filtration. This may be related to the pressure maintained during filtration.

In certain embodiments, the desired result is for a certain pressure to be maintained. For example, the desired result may be for a certain pressure to be maintained for a certain period of time. This may, for example, be measured by measuring the rate of pressure rise over a specified time period during filtration of each calibration sample for each concentration of filter aid tested.

Therefore, in certain embodiments, the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of filter aid used;

C) determining the minimum concentration of filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

In certain embodiments, the desired rate of pressure rise ranges from about 0.3 bar/hour to about 3 bar/hour. For example, the desired rate of pressure rise may range from about 0.3 bar/hour to about 2.5 bar/hour or from about 0.3 bar/hour to about 2 bar/hour or from about 0.3 bar/hour to about 1.5 bar/hour or from about 0.3 bar/hour to about 1 bar/hour or from about 0.3 bar/hour to about 0.6 bar/hour or from about 0.3 bar/hour to about 0.5 bar/hour. For example, the desired rate of pressure rise may range from about 1.5 bar/hour to about 3 bar/hour. For example, the desired rate of pressure rise may range from about 2 bar/hour to about 2.5 bar/hour. The desired rate of pressure rise may vary depending on the type of apparatus used.

In certain embodiments, the specified time period for measuring the pressure rate rise ranges from about 1 minute to about 2 hours. For example, the specified time period for measuring the pressure rate rise may range from about 2 minutes to about 105 minutes or from about 3 minutes to about 75 minutes or from about 4 minutes to about 60 minutes or from about 5 minutes to about 60 minutes or from about 10 minutes to about 60 minutes or from about 15 minutes to about 50 minutes or from about 20 minutes to about 40 minutes or from about 25 minutes to about 30 minutes.

In certain embodiments, a correction factor is applied to the concentration of filter aid that is selected for the filtration of a feed material. This may, for example, account for the differences between the feed material and calibration material.

In certain embodiments, the correction factor is calculated by filtering the feed material and the calibration material with one or more different concentration(s) of the test filter aid and comparing the difference in filtration of the feed material with the two or more different concentrations of test filter aid and the difference in filtration of the calibration material with the two or more different concentrations of test filter aid.

In certain embodiments, the correction factor F is defined by the following equation:

$$F = \frac{\Delta V_C}{\Delta V_T}$$

in which

$\Delta V_c$ is the difference in the volume of filtrate produced in the specified time period or time taken to obtain a specified amount of filtrate during filtration of a calibration sample of the material used to create the calibration curve with the first concentration and the second concentration of the test filter aid; and

$\Delta V_T$ is the difference in the volume of filtrate produced in the specified time period or time taken to obtain a specified amount of filtrate during filtration of the first test sample of feed material with the first concentration of filter aid and the second test sample of feed material with the second concentration of filter aid.

Where the correction factor F is defined by the equation specified above, the concentration of filter aid for filtration of the feed material that was determined by the method described above may be divided by the correction factor F.

In certain embodiments, the method for selecting the concentration of filter aid to use for the filtration of a feed material may therefore further comprise:

d) filtering a second test sample of the feed material with a second concentration of the test filter aid that is different to the first concentration of the test filter aid; and

e) measuring the volume of filtrate produced in the specified time period or measuring the time taken to obtain a specified amount of filtrate.

The difference in volume of filtrate produced in the specified time period or the difference in the time taken to obtain a specified amount of filtrate may then be calculated.

In certain embodiments, the persons performing the method for selecting the concentration of filter aid to use for the filtration of a feed material will be provided with the information relating to the difference in filtration of the calibration material with the two or more different concentrations of test filter aid, for example from an external source. In other embodiments, the persons performing the method for selecting the concentration of filter aid to use for the filtration of a feed material will calculate the

information relating to the difference in filtration of the calibration material with the two or more different concentrations of test filter aid themselves.

In certain embodiments, the method for selecting the concentration of filter aid to use for the filtration of a feed material may therefore further comprise:

- filtering a calibration material with a first concentration of a test filter aid and a second concentration of a test filter aid that is different to the first concentration; and
- measuring the volume of filtrate produced in the specified time period or measuring the time taken to obtain a specified amount of filtrate.

The difference in volume of filtrate produced in the specified time period or the difference in the time taken to obtain a specified amount of filtrate may then be calculated.

The type of test filter aid used (e.g. the permeability of the filter aid used and type of mineral etc.) for the filtration of the samples of feed material and calibration material is the same for all filtration tests for selecting the concentration of filter aid to use, creating the calibration curve and determining the correction factor.

The first concentration of test filter aid may be as described above. The first concentration of test filter aid is the same for the tests where samples of feed material and tests where samples of calibration material are filtered.

The second concentration of test filter aid is different to the first concentration of test filter aid. In certain embodiments, the second concentration is higher than the first concentration of test filter aid. In certain embodiments, the second concentration is lower than the first concentration of test filter aid. The difference between the first concentration of filter aid and second concentration of filter aid is at least about 50 g/HL. For example, the difference between the first concentration of filter aid and second concentration of filter aid may be at least about 75 g/HL or at least about 100 g/HL or at least about 125 g/HL or at least about 150 g/HL or at least about 175 g/HL or at least about 200 g/HL.

In certain embodiments, the second concentration of test filter aid ranges from about 100 g/HL to about 600 g/HL. For example, the second concentration of the test filter aid

may be equal to or greater than about 50 g/HL or equal to or greater than about 100 g/HL or equal to or greater than about 150 g/HL or equal to or greater than about 200 g/HL. For example, the second concentration of the test filter aid may be equal to or less than about 500 g/HL or equal to or less than about 450 g/HL or equal to or less than about 400 g/HL or equal to or less than about 350 g/HL or equal to or less than about 300 g/HL or equal to or less than about 250 g/HL.

The second concentration of the test filter aid may, for example, range from about 50 g/HL to about 400 g/HL, for example from about 100 g/HL to about 350 g/HL, for example from about 150 g/HI to about 300 g/HL, for example from about 150 g/HL to about 250 g/HL.

The concentration of calibration material in the calibration samples is the same for the filtrations with the first and second concentrations of the test filter. In certain embodiments, the concentration of calibration material ranges from about 1 g/L to about 5 g/L. For example, the concentration of calibration material may range from about 1.5 g/L to about 4.5 g/L or from about 2 g/L to about 4 g/L or from about 2.5 g/L to about 3.5 g/L. For example, the concentration of calibration material may be about 3 g/L.

Method for determining ease of filtration

It was further surprisingly found that the solutes present in a feed material also affect the filtration of the feed material. The degree to which the solutes present in a feed material affect its filtration can be determined by comparing the filtration of the feed material with particulate matter substantially removed and the filtration of water, for example distilled water. The particulate matter present in a feed material may be substantially removed by an initial filtration with a filter aid. This may be referred to as the solubles-imposed filtration flow loss (SIFFL).

There is therefore provided herein a method comprising:

a) filtering a first test sample of a feed material with a first concentration of a test filter aid;

b) filtering the filtrate produced in step a) with the same concentration of the test filter aid;

c) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

d) calculating the difference in volume of filtrate produced in the specified time period or the difference in the time taken for the specified amount of filtrate to be produced for the first test sample of the feed material (as measured in step c)) and water when filtered using the same concentration of the test filter aid.

Step a) substantially removes the particulate matter in the feed material. Therefore, step b) provides filtration information (e.g. volume of filtrate produced in a specified time period or time taken for a specified amount of filtrate to be produced) for the feed material with particulate matter substantially removed.

The relevant values for volume of filtrate produced in the specified time period or time taken for the specified amount of filtrate to be produced for water may be provided by an external source or may be measured by the person performing the method described herein.

Therefore, the method may further comprise measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced for a water test sample. For example, the method may further comprise filtering a sample of water with the same concentration of the test filter aid, and measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced. In certain embodiments, the volume of the first test sample of the feed material and the water test sample is the same.

Calculating the SIFFL is an indicator of the effect of solubles in the feed material on its filtration. The SIFFL may be an indicator of the ease of filtration of the feed material, where a lower SIFFL value indicates that the feed material will be easier to filter than a higher SIFFL value. Ease of filtration may, for example, relate to the speed of filtration or to the speed of pressure increase during filtration. A lower SIFFL value may indicate that the feed material will be quicker to filter than a higher SIFFL value. A lower SIFFL value may indicate that the rate of pressure rise during filtration will be lower than for a higher SIFFL value.

This may enable a manufacturing process involving filtration of the feed material to be designed with this in mind. For example, the manufacturing process may be designed to adjust the pH of the feed material and/or introduce an additive into the feed material and/or add an additional filtration step to the filtration process and/or to increase the length of time allocated for the filtration process.

The SIFFL may also enable comparison of one or more different feed materials. For example, feed materials with higher SIFFL values may be more difficult to filter than feed materials with lower SIFFL values.

The filter aid and conditions used for the method described herein may be in accordance with the filter aid and conditions used for a method for determining concentration of filter aid as described above.

The feed material may, for example, be any liquid that needs to be filtered. The feed material may, for example, be an aqueous or non-aqueous liquid.

The feed material may, for example, be any precursor to a non-edible oil or biodiesel. For example, the feed material may be a petroleum-based crude oil or derived from a petroleum-based crude oil. For example, the feed material may be a precursor to diesel.

Biodiesel is a form of purified alkyl esters of fatty acids generally referred to as fatty acid alkyl esters (FAAEs). Production of these FAAEs is achieved by the transesterification of animal and/or vegetable fats and/or oils or by the esterification of fatty acids, including free fatty acids (FFAs) found in degraded fat oil. Biodiesel can also be derived from triacylglycerides (also called triglycerides), which may be obtained from plant sources and/or animal fat sources. However, simply performing the esterification and/or transesterification of fatty acids is not enough to produce a usable biodiesel fuel. FFAEs contain impurities that can crystallize, foul engines, and cause numerous problems for the user. Crude biodiesel therefore may undergo filtration to obtain biodiesel that can be marketed commercially. The feed material may, for example, be crude biodiesel or any biodiesel precursor. The feed material may, for example, comprise, consist essentially of or consist of FFAEs.

The feed material may, for example, be any precursor to a food or beverage product. For example, the feed material may be a precursor of sugar syrups, beer, wine, liquor, milk, fruit juice, water or (edible) oils (e.g. animal oils or vegetable oils such as olive oil). For example, the feed material may be a precursor of beer or wine.

The first concentration of the test filter aid may, for example, range from about 100 g/HL to about 600 g/HL (HL = hectolitre = hl = hL). For example, the first concentration of the test filter aid may be equal to or greater than about 200 g/HL or equal to or greater than about 250 g/HL or equal to or greater than about 300 g/HL or equal to or greater than about 350 g/HL. For example, the first concentration of the test filter aid may be equal to or less than about 500 g/HL or equal to or less than about 450 g/HL.

The first concentration of the test filter aid may, for example, range from about 150 g/HL to about 550 g/HL, for example from about 200 g/HL to about 500 g/HL, for example from about 250 g/HL to about 450 g/HL, for example from about 300 g/HL to about 450 g/HL, for example from about 350 g/HL to about 450 g/HL. The first concentration of the test filter aid may, for example, range from about 360 g/HL to about 440 g/HL, for example from about 370 g/HL to about 430 g/HL, for example from about 380 g/HL to about 420 g/HL, for example from about 390 g/HL to about 410 g/HL, for example from about 395 g/HL to about 405 g/HL. For example, the first concentration of the test filter aid may be about 400 g/HL.

The test filter aid may, for example, be a mineral filter aid. The mineral filter aid may, for example, be diatomaceous earth, perlite or a combination thereof.

The test filter aid may, for example, be a low permeability filter aid. For example, the test filter aid may have a permeability ranging from 0 darcy to about 2 darcy. For example, the filter aid may have a permeability ranging from about 0.05 darcy to about 1.9 darcy, for example ranging from about 0.1 darcy to about 1.8 darcy, for example from about 0.15 darcy to about 1.7 darcy, for example from about 0.2 darcy to about 1.6 darcy, for example from about 0.25 darcy to about 1.5 darcy, for example from about 0.3 darcy to about 1.4 darcy, for example from about 0.35 darcy to about 1.3 darcy, for example from about 0.4 darcy to about 1.2 darcy, for example from about 0.45 darcy to about 1.1 darcy, for example from about 0.5 darcy to about 1.0 darcy. For example, the test filter aid may have a permeability ranging from about 0.01 darcy to about 1 darcy, for example from about 0.01 darcy to about 0.8 darcy, for example from

about 0.01 darcy to about 0.6 darcy, for example from about 0.01 darcy to about 0.5 darcy, for example from about 0.05 darcy to about 0.4 darcy, for example from about 0.1 darcy to about 0.3 darcy, for example from about 0.1 darcy to about 0.2 darcy.

In certain embodiments, the test filter aid is diatomaceous earth having a permeability ranging from about 0 darcy to about 1 darcy, for example from about 0.01 darcy to about 0.8 darcy, for example from about 0.01 darcy to about 0.6 darcy, for example from about 0.01 darcy to about 0.5 darcy, for example from about 0.05 darcy to about 0.4 darcy, for example from about 0.1 darcy to about 0.3 darcy, for example from about 0.1 darcy to about 0.2 darcy.

In certain embodiments, the test filter aid is perlite having a permeability ranging from about 1 darcy to about 2 darcy, for example from about 1.1 darcy to about 1.95 darcy, for example from about 1.2 darcy to about 1.9 darcy, for example from about 1.3 darcy to about 1.85 darcy, for example from about 1.4 darcy to about 1.8 darcy. In certain embodiments, the test filter aid is perlite having a permeability ranging from about 1.5 darcy to about 2 darcy or from about 1.6 darcy to about 1.9 darcy or from about 1.8 darcy to about 1.9 darcy.

Permeability may, for example, be measured by the water permeability method described by Darcy.

The specified period of time for measuring the volume of filtrate produced may be equal to or less than about 10 minutes. For example, the specified period of time for measuring the volume of filtrate produced may be equal to or less than about 9 minutes or equal to or less than about 8 minutes or equal to or less than about 7 minutes or equal to or less than about 6 minutes or equal to or less than about 5 minutes. The specified time period for measuring the volume of filtrate produced may be equal to or less than about 60 seconds. For example, the specified time period for measuring the volume of filtrate produced may be equal to or less than about 55 seconds or equal to or less than about 50 seconds or equal to or less than about 45 seconds or equal to or less than about 40 seconds or equal to or less than about 35 seconds or equal to or less than about 30 seconds or equal to or less than about 25 seconds or equal to or less than about 20 seconds or equal to or less than about 15 seconds. For example, the specified time period for measuring the volume of filtrate produced may be equal to or greater than about 5 seconds or equal to or greater than about 10 seconds or equal

to or greater than about 15 seconds. The specified period of time for measuring the volume of filtrate produced may, for example, be from about 5 seconds to about 60 seconds, for example from about 10 seconds to about 40 seconds, for example from about 15 seconds to about 30 seconds. The specified period of time for measuring the volume of filtrate produced may, for example, be about 30 seconds.

The specified volume of filtrate may, for example, be equal to or less than about 1000 mL. For example, the specified volume of filtrate may be equal to or less than about 900 mL or equal to or less than about 800 mL or equal to or less than about 700 mL or equal to or less than about 600 mL or equal to or less than about 500 mL. The specified volume of filtrate may, for example, be equal to or greater than about 50 mL, for example equal to or greater than about 100 mL or equal to or greater than about 150 mL or equal to or greater than about 200 mL or equal to or greater than about 250 mL. The specified volume of filtrate may, for example, be from about 50 mL to about 1000 mL, for example from about 100 mL to about 500 mL, for example from about 50 mL to about 250 mL.

The filtering of the test samples of the feed material with test filter aid may, for example, occur at a pressure equal to or less than about 1 bar. For example, the filtering of the test samples of the feed material with test filter aid may occur at a pressure equal to or less than about 0.9 bar or equal to or less than about 0.8 bar or equal to or less than about 0.7 bar or equal to or less than about 0.6 bar or equal to or less than about 0.5 bar or equal to or less than about 0.4 bar or equal to or less than about 0.3 bar or equal to or less than about 0.2 bar or equal to or less than about 0.1 bar. The filtering of the test samples of the feed material with test filter aid may, for example, occur at a pressure ranging from about 0.01 bar to about 1 bar, for example from about 0.05 bar to about 0.5 bar, for example from about 0.05 bar to about 0.3 bar, for example from about 0.05 bar to about 0.2 bar.

Method for selecting grade of filter aid

There is provided herein a method for selecting the permeability of filter aid to use for the filtration of a feed material. There is further provided herein a method for creating a calibration curve for selecting the permeability of filter aid to use for the filtration of a feed material. There is also provided herein a use of the calibration curve for selecting the permeability of filter aid to use for the filtration of a feed material.

The method for selecting the permeability of filter aid to use for the filtration of a feed material may select a permeability of filter aid for the filtration of the feed material that is within an optimum range for permeability of filter aid. The term “optimum” may, for example, refer to the permeability of filter aid that provides a desired clarity/turbidity of filtrate. The desired clarity may vary depending on the requirements of the manufacturer and the desired product.

The method for selecting the permeability of filter aid may comprise determining the difference in result obtained when filtering a feed material with one or more different filter aids having a different permeability and extrapolating the results to filter aids having other permeabilities. For example, the method for selecting the permeability of filter aid may comprise determining the difference in clarity achieved when filtering a feed material with one or more different filter aids having different permeabilities and extrapolating the results to filter aids having other permeabilities.

The method for selecting the permeability of filter aid comprises creating a calibration curve. The term “calibration curve” relates to a mathematical relationship between a particular characteristic of a calibration material and a preferred concentration of filter aid to use for filtration of the calibration material. Creation of the calibration curve may therefore comprise defining the relationship between the permeability of a filter aid and a particular characteristic of the feed material or filtrate, such as turbidity. This may or may not involve plotting a graph. The calibration curve may therefore be expressed as a mathematical formula or may be displayed by plotting on a graph.

The method for selecting the permeability of filter aid to use for the filtration of a feed material may, for example, comprise:

a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
b. measuring the turbidity of the first filtrate;
c. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
d. measuring the turbidity of the second filtrate;
e. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced;

f. selecting a desired turbidity of the feed material after filtration and using the relationship defined in step e to select the permeability of filter aid to use based on the desired turbidity of the feed material after filtration.

In certain embodiments, step e comprises creating a calibration curve by plotting the turbidity of each filtrate against the permeability of the filter aid used to produce the filtrate, wherein the permeability of the filter aid is plotted on a logarithmic scale, and determining the equation for the line of best fit. The line of best fit may, for example, be determined by drawing a straight line between each point.

The method for selecting the permeability of filter aid to use for the filtration of a feed material may, for example, comprise filtering one or more further test samples of the feed material with one or more further filter aids having different permeabilities and measuring the turbidity of the filtrate produced. The results obtained can then be used to define the relationship between the permeability of filter aid and turbidity of the filtrate produced. For example, the results obtained can also be plotted and the line of best fit determined in order to create a calibration curve.

The filter aid may, for example, be a mineral filter aid. The mineral filter aid may, for example, be diatomaceous earth, perlite or a combination thereof.

In certain embodiments, the one or more filter aids that are used in the method for selecting the permeability of filter aid to use for filtration of a feed are the same type of filter aid, for example are all the same type of mineral such as all diatomaceous earth filter aids or all perlite filter aids.

In certain embodiments, the first filter aid is a low permeability filter aid. In certain embodiments, the first filter aid has a permeability ranging from about 0 to about 3 darcy. For example, the first filter aid may have a permeability ranging from about 0.01 darcy to about 2.5 darcy or from about 0.1 darcy to about 2 darcy or from about 0.5 darcy to about 1.9 darcy or from about 1 darcy to about 1.8 darcy. For example, the first filter aid may have a permeability ranging from about 0.01 darcy to about 1.5 darcy or from about 0.1 darcy to about 1 darcy or from about 0.1 darcy to about 0.5 darcy.

In certain embodiments, the second filter aid is a high permeability filter aid. In certain embodiments, the second filter aid has a permeability ranging from about 3 darcy to

about 20 darcy. For example, the second filter aid may have a permeability ranging from about 3 darcy to about 18 darcy or from about 3 darcy to about 16 darcy or from about 3 darcy to about 15 darcy or from about 3 darcy to about 14 darcy or from about 3 darcy to about 12 darcy or from about 3 darcy to about 10 darcy or from about 3 darcy to about 8 darcy or from about 3 darcy to about 6 darcy or from about 3 darcy to about 5 darcy. For example, the second filter aid may have a permeability ranging from about 3 darcy to about 6 darcy or from about 3.5 darcy to about 5.5 darcy or from about 3.5 darcy to about 5 darcy or from about 3.5 darcy to about 4.5 darcy. For example, the second filter aid may have a permeability of about 4 darcy.

In certain embodiments, the difference in permeability of the filter aid having the lowest permeability (e.g. the first filter aid) and the filter aid having the highest permeability (e.g. the second filter aid) is at least about 2 darcy. For example, the difference in permeability of the filter aid having the lowest permeability and the filter aid having the highest permeability may be at least about 2.5 darcy or at least about 3 darcy or at least about 3.5 darcy or at least about 4 darcy. For example, the difference in permeability of the filter aid having the lowest permeability and the filter aid having the highest permeability may be equal to or less than about 15 darcy, for example equal to or less than about 12 darcy, for example equal to or less than about 10 darcy, for example equal to or less than about 8 darcy, for example equal to or less than about 6 darcy.

The concentration of the various filter aids tested in the method for selecting the permeability of filter aid to use for the filtration of a feed material is the same.

The concentration of the filter aids tested may, for example, range from about 100 g/HL to about 600 g/HL. For example, the first concentration of the test filter aid may be equal to or greater than about 100 g/HL or equal to or greater than about 150 g/HL or equal to or greater than about 200 g/HL or equal to or greater than about 250 g/HL or equal to or greater than about 300 g/HL or equal to or greater than about 350 g/HL. For example, the first concentration of the test filter aid may be equal to or less than about 500 g/HL or equal to or less than about 450 g/HL.

The turbidity of the filtrate may, for example, be measured after a certain volume has been collected or after a certain period of time has passed. For example, the turbidity

of the filtrate may be measured after a certain volume has been collected or after a certain period of time has passed, whichever occurs first.

The certain volume collected may, for example, range from about 200 ml to about 600 ml. For example, the certain volume collected may range from about 250 ml to about 550 ml or from about 300 ml to about 500 ml or from about 450 ml to about 450 ml.

The certain period of time may, for example, range from about 60 seconds to about 180 seconds. For example, the certain period of time may range from about 75 seconds to about 165 seconds or from about 90 seconds to about 150 seconds or from about 105 seconds to about 135 seconds.

The filtering of the test samples of the feed material with a filter aid may, for example, occur at a pressure equal to or less than about 1 bar. For example, the filtering of the test samples of the feed material with a filter aid may be equal to or less than about 0.9 bar or equal to or less than about 0.8 bar or equal to or less than about 0.7 bar or equal to or less than about 0.6 bar or equal to or less than about 0.5 bar or equal to or less than about 0.4 bar or equal to or less than about 0.3 bar or equal to or less than about 0.2 bar or equal to or less than about 0.1 bar.

Turbidity is a measure of the clarity (cloudiness or haziness) of the filtrate. Turbidity may, for example, be measured by the propensity of particles to scatter a light beam. This may use an instrument called a nephelometer with a detector set up to the side of the light beam. More light reaches the detector if there are lots of particles scattering the source beam compared to fewer particles. The units of turbidity from a nephelometer are Nephelometric Turbidity Units (NTU).

The feed material and/or calibration material may, for example, be as described herein.

Apparatus used for filtration tests

There is further provided herein a filtration device suitable for any one or more of the methods described herein and the use of said filtration device for said methods.

The filtration device comprises a container that is separated into at least two sections by a semi-permeable barrier.

The semi-permeable barrier may, for example, have openings having a diameter ranging from about 20 µm to about 80 µm, for example from about 25 µm to about 75 µm, for example from about 30 µm to about 70 µm, for example from about 35 µm to about 65 µm, for example from about 40 µm to about 60 µm. The size of the openings may be chosen depending on the particle size of the filter aid to be used with the apparatus.

The semi-permeable barrier may, for example, be paper. The semi-permeable barrier may, for example, be a woven screen, for example a metal woven screen, for example a stainless steel woven screen.

The container of the filtration device may be or may be capable of being pressurized. For example, the container of the filtration device may be or may be capable of being pressurized to a pressure equal to or less than about 1 bar, for example equal to or less than about 0.9 bar, for example equal to or less than about 0.8 bar, for example equal to or less than about 0.7 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.5 bar, for example equal to or less than about 0.4 bar, for example equal to or less than about 0.3 bar, for example equal to or less than about 0.2 bar, for example equal to or less than about 0.1 bar. The container may be pressurized using a gas cartridge, for example a nitrogen or carbon dioxide gas cartridge.

During use of the filtration device, the feed material may be introduced into one section of the container and the filtrate recovered from the other section of the container.

## EXAMPLES

The following illustrates examples of the methods and other aspects described herein. Thus, these Examples should not be considered as limitations of the present disclosure, but are merely in place to teach how to make examples of the present disclosure.

**Example 1 – calibration curve creation**

Ovaltine® obtained in the UK (the calibration material) was mixed with water to produce a variety of calibration samples having different concentrations (between 1 g/L and 12 g/L at 1 g/L increments).

The calibration samples (500 mL) were filtered in a filtration device using 2 g (400 g/HL) of a diatomaceous earth filter aid having a permeability of 0.18 darcy. The filtration device was in accordance with the filtration device shown in Figure 2 and comprised a vertical cylindrical pressure vessel fitted with a screw-release screen (Whatman 541, 47 mm diameter) in the base, into which a filter paper was inserted before each calibration sample was filtered. The filter paper acts similarly to a first pre-coat layer in an industrial filter device. The apparatus was pressurised from a nitrogen cylinder (0.6 bar) to provide the driving force across the filter septum.

The volume of filtrate produced in the first 30 seconds was measured for each calibration sample. A linear relationship between the volume of filtrate produced in the first 30 seconds and the concentration of the calibration sample was observed.

The preferred concentration of the filter aid to use for filtration of three different calibration samples of different concentrations (5 g/L, 6g/L and 9.5 g/L) was determined by filtering samples with different concentrations of the filter aid (100 g/HL, 130 g/HL, 200 g/HL, 250 g/HL and 300 g/HL) using a Walton Filter at a flow rate of 9 HL/$m^2$/h (30 mL/min in the Walton Filter). The preferred concentration of filter aid under these conditions is the minimum concentration of filter aid that results in a rate of pressure rise in the order of 2.0 to 2.5 bar/h. The rate of pressure rise [bar/h] was measured for each sample over a time period of 10-40 min and the results are shown in the following table:

| Concentration of filter aid (SSC) [g/HL] | 5 g/L sample of Ovaltine | 6 g/L sample of Ovaltine | 9.5 g/L sample of Ovaltine |
|---|---|---|---|
| | 3.69 | | |
| 100 | 2.5/2.06 | 3.36/3.75 | |
| 130 | | 2.20 | |
| 200 | 0.60 | 0.93 | 3.90 |
| 250 | | | 2.38 |
| 400 | 0.36 | | 1.3 |

A calibration curve was created by plotting the preferred concentration of the filter aid determined from the pressure rise against the volume of filtrate produced in the first 30 seconds of filtration for each of the samples (see Figure 1). The equation for the line of best fit was then determined.

**Example 2 – selection of concentration of filter aid**

*Red wine 1 (turbidity of 19 ntu)*

A 500 mL test sample of red wine 1 was filtered in the filtration device using 2 g (400 g/HL) of the diatomaceous earth and filtration device used in Example 1 at an operating pressure of 0.6 bar. The volume of filtrate produced in the first 30 seconds was 212 mL. The concentration of Ovaltine that produced 212 mL of filtrate in 30 seconds (the Ovaltine equivalent value) was 6.0 g/L. From this, and the calibration curve determined in Example 1, the amount of filter aid to be used in filtering red wine 1 is selected as 250 g/HL.

To check that this assessment is correct, red wine 1 was also filtered on a Walton Filter at three different concentrations of SSC filter aid with the rate of pressure rise indicating the preferred amount of filter aid, the preferred rate of pressure rise being between 2 and 2.5 bar/h.

| Concentration of filter aid (SSC) [g/HL] | Rate of pressure rise of red wine 1 (19 ntu) (bar/h) |
|---|---|
| 100 | 4.56 |
| 200 | 2.20 |
| 400 | 1.10 |

From these results, it is clear that the preferred amount of filter aid is in the region of 250 g/HL, which is in agreement with the amount determined by using the calibration curve.

*Red wine 2 (turbidity of 370 ntu)*

A 500 mL test sample of red wine 2 was filtered using 2g (400 g/HL) of the diatomaceous earth and filtration device used in Example 1 at an operating pressure of 0.6 bar. The volume of filtrate produced in the first 30 seconds was 133 mL. The concentration of Ovaltine that produced 133 mL of filtrate in 30 seconds (the Ovaltine equivalent value) was 9.5 g/L. From this, and the calibration curve determined in Example 1, it can be determined that the preferred amount of filter aid to be used in filtering red wine 1 is 400 g/HL.

To check that this assessment is correct, red wine 2 was also filtered on a Walton Filter at three different concentrations of SSC filter aid with the rate of pressure rise indicating the preferred amount of filter aid, the preferred rate of pressure rise being between 2 and 2.5 bar/h.

| Concentration of filter aid (SSC) [g/HL] | Rate of pressure rise of red wine 2 (370 ntu) (bar/h) |
|---|---|
| 100 | |
| 200 | 6.60 |
| 400 | 1.80 |

From these results, it is clear that the preferred amount of filter aid is in the region of 400 g/HL or a little less, which is reasonably in agreement with the amount determined by using the calibration curve.

**Example 3 - selection of concentration of filter aid including use of a correction factor**

A 500 mL first test sample of local unfiltered beer from St Austell Brewery (82.7 ntu), characterised by a relatively high yeast content but generally lacking in any finer solids was filtered in the filtration device used in Example 1 using 2 g of the diatomaceous earth used in example 1 (400 g/HL) at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 15 seconds was 112 mL. Using a calibration curve created in accordance with the methods described herein, the preferred amount of filter aid to use for this beer is 148 g/HL.

To determine the correction factor, a second test sample of the beer was filtered in the filtration device used in Example 1 using 1 g of the diatomaceous earth used in example 1 (200 g/HL) at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 15 seconds was 127 mL.

The calibration material Ovaltine® obtained from the UK (3 g/L) was also filtered at concentrations of 200 g/HL and 400 g/HL of the diatomaceous earth used in example 1 in order to determine the correction factor.

| | First test sample (400 g/HL of SSC) [mL] | Second test sample (200 g/HL of SSC) [mL] | $\Delta V$ [mL] |
|---|---|---|---|
| Ovaltine at 3 g/L | 109 | 135 | −26 |
| Beer | 112 | 127 | −15 |

The correction factor, F, was calculated using the following equation:

$$F = \frac{\Delta V_C}{\Delta V_T}$$

in which

$\Delta V_c$ is the difference in the volume of filtrate formed in 15 seconds from the filtration of Ovaltine (3 g/L) with 400 g/HL and 200 g/HL of diatomaceous earth; and

$\Delta V_T$ is the difference in the volume of filtrate formed in 15 seconds from the filtration of the beer with 400 g/HL and 200 g/HL of diatomaceous earth.

$$F = \frac{\Delta V_C}{\Delta V_T} = \frac{-26}{-15} = 1.73$$

The preferred concentration of filter aid is then corrected by applying the correction factor to the predicted concentration of filter aid resulting from the filtration of the first test sample by dividing the initially predicted concentration of the filter aid by the correction factor.

$$\frac{148\ \mathrm{g/HL}}{1.73} = 85\ \mathrm{g/HL}$$

The selected concentration of filter aid to use for filtration of the beer is therefore 85 g/HL.

**Example 4 – selection of permeability of filter aid**

A 500 mL first test sample was filtered using 2 g of the diatomaceous earth used in example 1 in the filtration device used in Example 1 at an operating pressure of 0.1 bar and a 48 µm stainless steel weave screen. After either 2 minutes of filtering or after 400 mL of filtrate collected, a sample of filtrate was taken and placed in a cuvette. The turbidity of the filtrate was measured as 0.32 ntu.

A second 500 mL test sample was filtered using a diatomaceous earth having a permeability of 4.2 darcy in the filtration device used in Example 1 at an operating pressure of 0.1 bar and a 48 µm stainless steel weave screen. After either 2 minutes of filtering or after 400 mL of filtrate collected, a sample of filtrate was taken and placed in a cuvette. The turbidity of the filtrate was measured as 0.75 ntu.

These two turbidities (in ntu) were plotted against the permeabilities of the respective filter aids, wherein permeability was plotted on a logarithmic scale. The two points were connected with a straight line.

The appropriate permeability of filter aid can then be selected by selecting a particular desired turbidity.

**Example 5 – calibration curve creating using PIFFL**

Ovaltine® obtained in the UK (the calibration material) was mixed with water to produce a variety of calibration samples having different concentrations (between 1 g/L and 11 g/L at 1 g/L increments).

The calibration samples (500 mL) were filtered in a filtration device using 2 g (400 g/HL) of a diatomaceous earth filter aid having a permeability of 0.18 darcy. The filtration device comprised a vertical cylindrical pressure vessel fitted with a mesh fine enough to retain the filter aid. The mesh acts similarly to a first pre-coat layer in an industrial filter device. The apparatus was pressurised from a nitrogen cylinder (0.1 bar) to provide the driving force across the filter septum.

The volume of filtrate produced in the first 30 seconds was measured for each calibration sample.

All the filtrate of each calibration sample was collected and then this filtrate was filtered in the same filtration device using 2 g (400 g/HL) of a fresh sample of the same diatomaceous earth filter aid having a permeability of 0.18 darcy.

The volume of filtrate produced in the first 30 seconds was measured for each calibration sample.

The difference in volume of filtrate produced (PIFFL) was calculated. The results are shown in the following table:

| Original feed material Ovaltine® (g/L) | Volume of filtrate collected in first 30 seconds for original feed material (mL) | Volume of filtrate collected in first 30 seconds for filtrate of original feed material (mL) | PIFFL (mL) | Preferred concentration of filter aid (g/HL) |
|---|---|---|---|---|
| 1 | 156 | 162 | 6 | 40 |
| 2 | 131 | 156 | 25 | 80 |
| 3 | 112 | 160 | 48 | 120 |
| 4 | 95 | 157 | 62 | 160 |
| 5 | 87 | 154 | 67 | 200 |
| 6 | 78 | 154 | 76 | 240 |
| 7 | 69 | 145 | 76 | 280 |
| 8 | 62 | 150 | 88 | 320 |
| 9 | 56 | 151 | 95 | 360 |
| 10 | 50 | 143 | 93 | 400 |
| 11 | 46 | 149 | 103 | 440 |

A calibration curve was created by plotting the preferred concentration of the filter aid against the PIFFL value instead of the volume of filtrate produced in the first 30 seconds of filtration for each of the samples. The equation for the line of best fit was then determined. The preferred concentration of filter aid for each tested concentration of Ovaltine® was calculated using the preferred concentrations determined in Example 1 and based on the estimation that the preferred filter aid concentration for 10 g/L of Ovaltine® is 400 g/HL with a linear relationship (see right-hand column of the table above).

**Example 6 – selection of concentration of filter aid using PIFFL**

A 500 mL test sample of a beer from St. Austell Brewery (45 ntu), a decanted red wine (5.0 ntu), a white wine (254 ntu) or a 5x dilution of a red must (0.36 ntu) were each filtered using 2 g (400 g/HL) of the diatomaceous earth and a filtration device comprising a vertical cylindrical pressure vessel fitted with a mesh fine enough to retain

the filter aid, at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 30 seconds was measured.

All of the filtrate of each test sample was collected and each filtrate was then filtered in the filtration device using a 2 g (400 g/HL) fresh sample of the diatomaceous earth and filtration device used in Example 1 at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 30 seconds was measured.

The difference in volume of filtrate produced in the first 30 seconds for the original test sample and the filtrate of the respective test sample was calculated (PIFFL). The preferred amount of filter aid was determined using the calibration curve obtained in Example 5 above. The results are shown in the Table below.

| **Test Sample** | **Volume of filtrate collected in first 30 seconds for original test sample (mL)** | **Volume of filtrate collected in first 30 seconds for filtrate of original test sample (mL)** | **PIFFL (mL)** | **Preferred concentration of filter aid (g/HL)** |
|---|---|---|---|---|
| Beer (45 ntu) | 103 | 119 | 16 | 55 |
| Red Wine (5.0 ntu) | 95 | 131 | 36 | 90 |
| White wine (254 ntu) | 75 | 119 | 44 | 110 |
| Red must conc. 5x diluted (0.35 ntu) | 118 | 123 | 5 | 40 |

Standard Walton filter tests were performed on the beer and white wine and it was found that the predicted concentration of filter aid using the Walton filter test was very close to the predicted values in the table above.

**Example 7 – selection of concentration of filter aid using PIFFL**

A 500 mL test sample of a beer from Coors brewery before and after centrifugation, a beer from a Heineken brewery before and after centrifugation and a cider from a Heineken brewery were each filtered using 2 g (400 g/HL) of the diatomaceous earth and filtration device comprising a vertical cylindrical pressure vessel fitted with a mesh fine enough to retain the filter aid, at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 30 seconds was measured.

All of the filtrate of each test sample was collected and each filtrate was then filtered in the filtration device using a 2 g (400 g/HL) fresh sample of the diatomaceous earth and filtration device used in Example 1 at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 30 seconds was measured.

The difference in volume of filtrate produced in the first 30 seconds for the original test sample and the filtrate of the respective test sample was calculated (PIFFL). The preferred amount of filter aid was determined using the calibration curve obtained in Example 5 above.

The results are shown in the Table below.

| Test Sample | Volume of filtrate collected in first 30 seconds for original test sample (mL) | Volume of filtrate collected in first 30 seconds for filtrate of original test sample (mL) | PIFFL (mL) | Preferred concentration of filter aid (g/HL) |
|---|---|---|---|---|
| Coors beer after centrifuge | 76 | 87 | 11 | 53 |
| Coors beer before centrifuge | 60 | 86 | 26 | 72 |
| Heineken cider | 99 | 104 | 5 | 40 |
| Heineken beer after centrifuge | 93 | 97 | 4 | 40 |
| Heineken beer before centrifuge | 85 | 101 | 16 | 68 |

Standard Walton filter tests were performed and it was found that the predicted concentration of filter aid using the Walton filter test was very close to the predicted values in the table above.

**Example 8 – calculating of SIFFL**

A 500 mL sample of water was filtered using 2 g (400 g/HL) of the diatomaceous earth and filtration device comprising a vertical cylindrical pressure vessel fitted with a mesh fine enough to retain the filter aid, at an operating pressure of 0.1 bar. The volume of filtrate produced in the first 30 seconds was measured and found to be 162 mL.

The SIFFL value for each of the test samples tested in Example 7 was calculated using the values for volume of filtrate collected in the first 30 seconds for the filtrate of the original test sample. The results are shown in the table below.

| Test Sample | SIFFL |
|---|---|
| Coors beer after centrifuge | 75 |
| Coors beer before centrifuge | 76 |
| Heineken cider | 58 |
| Heineken beer after centrifuge | 65 |
| Heineken beer before centrifuge | 61 |

The foregoing broadly describes certain embodiments of the present invention without limitation. Variations and modifications as will be readily apparent to those skilled in the art are intended to be within the scope of the present invention as defined in and by the appended claims.

The following numbered paragraphs define particular embodiments of the present invention.

1. A method for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:
    a) filtering a first test sample of the feed material with a first concentration of a test filter aid;
    b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;
    c) using a calibration curve to select the concentration of filter aid to use based on the volume of filtrate produced or time taken measured in step b).

2. The method of paragraph 1, wherein the method further comprises:
    d) filtering a second test sample of the feed material with a second concentration of the test filter aid that is different to the first concentration of the test filter aid;

e) measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced;

f) determining a correction factor, F, by the following equation:

$$F = \frac{\Delta V_C}{\Delta V_T}$$

in which

$\Delta V_c$ is the difference in the volume of filtrate produced in the specified time period or time taken to obtain a specified amount of filtrate, during filtration of a calibration sample of the material used to create the calibration curve with the first concentration and the second concentration of the test filter aid; and

$\Delta V_T$ is the difference in the volume of filtrate produced in the specified time period or time taken to obtain a specified amount of filtrate, during filtration of the first test sample of the feed material with the first concentration of filter aid and the second test sample of the feed material with the second concentration of filter aid;

g) dividing the concentration of filter aid determined by the method of paragraph 1 by the correction factor, F.

3. The method of paragraph 1 or 2, wherein step c) includes:

(i) equating the feed material with a certain concentration of a calibration material by selecting the concentration of calibration material that produces the same volume of filtrate when filtered with the first concentration of the test filter aid in the specified time period or by selecting the concentration of calibration material takes the same amount of time to filter a specified amount of filtrate using the first concentration of the test filter aid; and

(ii) selecting the concentration of filter aid to use by selecting a preferred concentration of filter aid that is associated with the concentration of calibration material that has been equated with the feed material.

4. The method of any one of paragraphs 1 to 3, wherein the calibration curve was obtained by a method comprising:

i) filtering a plurality of calibration samples of a calibration material with the first concentration of the test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples;

iv) defining the relationship between the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii).

5. The method of paragraph 4, wherein the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of test filter aid used;

C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the test filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

6. The method of any one of paragraphs 1 to 5, wherein the filter aid is a mineral filter aid.

7. The method of paragraph 6, wherein the mineral filter aid is diatomaceous earth and/or perlite.

8. The method of any one of paragraphs 1 to 7, wherein the filter aid is a low permeability filter aid, for example having a permeability between 0 and about 2 Darcy.

9. The method of any one of paragraphs 1 to 8, wherein the first concentration of the test filter aid and/or the second concentration of the test filter aid ranges from about 100 g/HL to about 600 g/HL.

10. The method of any one of paragraphs 1 to 9, wherein the first concentration of the filter aid is equal to or greater than about 300 g/HL, for example equal to or greater than about 350 g/HL.

11. The method of any one of paragraphs 1 to 10, wherein the first concentration of the filter aid is equal to or less than about 500 g/HL, for example equal to or less than about 450 g/HL.

12. The method of any one of paragraphs 1 to 11, wherein the feed material is a precursor in wine or beer production.

13. The method of any one of paragraphs 1 to 12, wherein the specified time period for measuring the volume of filtrate produced is equal to or less than about 60 seconds, for example equal to or less than about 30 seconds, for example equal to or less than about 15 seconds.

14. The method of any one of paragraphs 1 to 13, wherein the filtering of the test samples of the feed material and/or plurality of calibration samples of a calibration material with a first concentration of a test filter aid occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

15. The method of any one of paragraphs 2 to 15, wherein the difference between the first concentration and the second concentration of the filter aid is at least about 50 g/HL, for example at least about 100 g/HL.

16. The method of any one of paragraphs 2 to 16, wherein the second concentration of the filter aid is less than about 300 g/HL, for example equal to or less than about 250 g/HL.

17. The method of any one of paragraphs 2 to 17, wherein the second concentration of the filter aid is equal to or greater than about 100 g/HL, for example equal to or greater than about 150 g/HL.

18. The method of any one of paragraphs 1 to 17, wherein the calibration material comprises organic solids, for example compressible organic solids.

19. The method of any one of paragraphs 1 to 18, wherein the calibration material comprises barley malt extract.

20. The method of any one of paragraphs 5 to 19, wherein the desired pressure rate rise ranges from about 2 bar/hour to about 2.5 bar/hour.

21. The method of any one of paragraphs 5 to 20, wherein the specified time period for measuring the pressure rate rise ranges from about 1 minute to about 2 hours, for example from about 5 minutes to about 1 hour.

22. A method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples;

iv) defining the relationship between the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii).

23. The method of paragraph 22, wherein the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of filter aid used;

C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

24. The method of paragraph 22 or 23, wherein the filter aid is a mineral filter aid.

25. The method of paragraph 24, wherein the mineral filter aid is diatomaceous earth and/or perlite.

26. The method of any one of paragraphs 22 to 25, wherein the filter aid is a low permeability filter aid, for example having a permeability between 0 and about 2 Darcy.

27. The method of any one of paragraphs 22 to 26, wherein the first concentration of the test filter aid ranges from about 100 g/HL to about 600 g/HL.

28. The method of any one of paragraphs 22 to 27, wherein the first concentration of the test filter aid is equal to or greater than about 300 g/HL, for example equal to or greater than about 350 g/HL.

29. The method of any one of paragraphs 22 to 28, wherein the first concentration of the test filter aid is equal to or less than about 500 g/HL, for example equal to or less than about 450 g/HL.

30. The method of any one of paragraphs 22 to 29, wherein the feed material is a precursor in wine or beer production.

31. The method of any one of paragraphs 22 to 30, wherein the specified time period for measuring the volume of filtrate produced is equal to or less than about 60 seconds, for example equal to or less than about 30 seconds, for example equal to or less than about 15 seconds.

32. The method of any one of paragraphs 22 to 31, wherein the filtering of the plurality of calibration samples of a calibration material with a first concentration of a test filter aid occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

33. The method of any one of paragraphs 22 to 32, wherein the calibration material comprises organic solids, for example compressible organic solids.

34. The method of any one of paragraphs 22 to 33, wherein the calibration material comprises barley malt extract.

35. The method of any one of paragraphs 22 to 34, wherein the desired rate of pressure rise ranges from about 2 bar/hour to about 2.5 bar/hour.

36. The method of any one of paragraphs 22 to 35, wherein the specified time period for measuring the pressure rate rise ranges from about 1 minute to about 2 hours, for example from about 5 minutes to about 1 hour.

37. A method for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising:
    a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
    b. measuring the turbidity of the first filtrate;
    c. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
    d. measuring the turbidity of the second filtrate;
    e. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced;
    f. selecting a desired turbidity of the feed material after filtration and using the calibration curve to select the permeability of filter aid to use based on the desired turbidity of the feed material after filtration.

38. The method of paragraph 37, wherein the method further comprises:
    filtering one or more further test samples of the feed material with one or more further filter aids having different permeabilities to the first and second filter aids to produce one or more further filtrates;
    measuring the turbidity of the one or more further filtrates;
    using the turbidity of the one or more further filtrate(s) to define the relationship between the permeability of filter aid and turbidity of the filtrate produced.

39. The method of paragraph 37 or 38, wherein one or more of the filter aid(s) is a mineral filter aid.

40. The method of paragraph 39, wherein the mineral filter aid is diatomaceous earth and/or perlite.

41. The method of any one of paragraphs 37 to 40, wherein all of the filter aids used in the method are the same type of mineral, for example wherein all of the filter aids are diatomaceous earth or wherein all of the filter aids are perlite.

42. The method of any one of paragraphs 37 to 41, wherein the first filter aid has a permeability ranging from about 0.01 to about 2 darcy, for example from about 0.1 to about 0.5 darcy.

43. The method of any one of paragraphs 37 to 42, wherein the second filter aid has a permeability ranging from about 3 to about 6 darcy, for example from about 3.5 to about 4.5 darcy.

44. The method of any one of paragraphs 37 to 43, wherein the permeability of the filter aid having the highest permeability differs from the permeability of the filter aid having the lowest permeability by at least about 2 darcy, for example at least about 3 darcy.

45. The method of any one of paragraphs 37 to 44, wherein the first filter aid is a low permeability filter aid, for example having a permeability ranging from 0 to about 2 Darcy.

46. The method of any one of paragraphs 37 to 45, wherein the second filter aid is a high permeability filter aid, for example having a permeability greater than about 2 Darcy and equal to or less than about 20 Darcy.

47. The method any one of paragraphs 37 to 46, wherein the concentration of the first filter aid and the second filter aid is the same.

48. The method of any one of paragraphs 37 to 47, wherein the concentration of the first filter aid and the second filter aid ranges from about 100 g/HL to about 600 g/HL.

49. The method of any one of paragraphs 37 to 48, wherein the feed material is a precursor in wine or beer production.

50. The method of any one of paragraphs 37 to 49, wherein the turbidity of the filtrate is measured after a certain volume has been collected or after a certain period of time has passed.

51. The method of paragraph 50, wherein the certain volume ranges from about 200 ml to about 600 ml, for example from about 300 ml to about 500 ml.

52. The method of paragraph 50 or 51, wherein the certain period of time ranges from about 60 seconds to about 180 seconds, for example from about 90 seconds to about 150 seconds.

53. The method of any one of paragraphs 50 to 52, wherein the turbidity of the filtrate is measured after a certain volume has been collected or after a certain period of time has passed, whichever occurs first.

54. The method of any one of paragraphs 37 to 53, wherein the filtering of the test samples of the feed material occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

55. A method for creating a calibration curve for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising:
    a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
    b. measuring the turbidity of the first filtrate;
    c. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
    d. measuring the turbidity of the second filtrate;
    e. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced;

56. The method of paragraph 55, wherein the method further comprises:

filtering one or more further test samples of the feed material with one or more further filter aids having different permeabilities to the first and second filter aids to produce one or more further filtrates;

measuring the turbidity of the one or more further filtrates;

using the turbidity of the one or more further filtrate(s to define the relationship between the permeability of filter aid and turbidity of the filtrate produced.

57. The method of paragraph 55 or 56, wherein one or more of the filter aid(s) is a mineral filter aid.

58. The method of paragraph 57, wherein the mineral filter aid is diatomaceous earth and/or perlite.

59. The method of any one of paragraphs 55 to 58, wherein all of the filter aids used in the method are the same type of mineral, for example wherein all of the filter aids are diatomaceous earth or wherein all of the filter aids are perlite.

60. The method of any one of paragraphs 55 to 59, wherein the first filter aid has a permeability ranging from about 0.01 to about 1 darcy, for example from about 0.1 to about 0.5 darcy.

61. The method of any one of paragraphs 55 to 60, wherein the second filter aid has a permeability ranging from about 3 to about 5 darcy, for example from about 3.5 to about 4.5 darcy.

62. The method of any one of paragraphs 55 to 61, wherein the permeability of the filter aid having the highest permeability differs from the permeability of the filter aid having the lowest permeability by at least about 2 darcy, for example at least about 3 darcy.

63. The method of any one of paragraphs 55 to 62, wherein the first filter aid is a low permeability filter aid, for example having a permeability ranging from 0 to about 2 darcy.

64. The method of any one of paragraphs 55 to 63, wherein the second filter aid is a high permeability filter aid, for example having a permeability greater than about 2 Darcy and equal to or less than about 20 Darcy.

65. The method any one of paragraphs 55 to 64, wherein the concentration of the first filter aid and the second filter aid is the same.

66. The method of any one of paragraphs 55 to 65, wherein the concentration of the first filter aid and the second filter aid ranges from about 100 g/HL to about 600 g/HL.

67. The method of any one of paragraphs 55 to 66, wherein the feed material is a precursor in wine or beer production.

68. The method of any one of paragraphs 55 to 67, wherein the turbidity of the filtrate is measured after a certain volume has been collected or after a certain period of time.

69. The method of paragraph 68, wherein the certain volume ranges from about 200 ml to about 600 ml, for example from about 300 ml to about 500 ml.

70. The method of paragraph 68 or 69, wherein the certain period of time ranges from about 60 seconds to about 180 seconds, for example from about 90 seconds to about 150 seconds.

71. The method of any one of paragraphs 68 to 70, wherein the turbidity of the filtrate is measured after a certain volume has been collected or after a certain period of time has passed, whichever occurs first.

72. The method of any one of paragraphs 55 to 71, wherein the filtering of the test samples of the feed material occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

73. A method for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

a) filtering a first test sample of the feed material with a first concentration of a test filter aid;

b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;

c) filtering the filtrate produced by step a) with the same concentration of the test filter aid;

d) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced;

e) calculating the difference in volume of filtrate produced or time taken measured in steps b) and d),

f) using a calibration curve to select the concentration of filter aid to use based on the difference in volume of filtrate produced or time taken calculated in step e).

74. The method of paragraph 73, wherein step f) includes:

(iii) equating the feed material with a certain concentration of a calibration material by selecting the concentration of calibration material that has the same difference in volume of filtrate produced in the specified time period or the same difference in time taken to produce a specified volume of filtrate when the original calibration sample and the filtrate of the calibration sample are filtered with the first concentration of the test filter aid; and

(iv) selecting the concentration of filter aid to use by selecting a preferred concentration of filter aid that is associated with the concentration of calibration material that has been equated with the feed material.

75. The method of any one of paragraphs 73 or 74, wherein the calibration curve was obtained by a method comprising:

i) filtering a plurality of calibration samples of a calibration material with the first concentration of the test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample;

iii) filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid;

iv) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample;

v) calculating the difference in volume of filtrate produced or time taken measured in steps ii) and iv);

vi) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples; and

vii) defining the relationship between the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced or time taken calculated in step v).

76. The method of paragraph 75, wherein the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of test filter aid used;

C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the test filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

77. The method of any one of paragraphs 73 to 76, wherein the filter aid is a mineral filter aid.

78. The method of paragraph 77, wherein the mineral filter aid is diatomaceous earth and/or perlite.

79. The method of any one of paragraphs 73 to 78, wherein the filter aid is a low permeability filter aid, for example having a permeability between 0 and about 2 Darcy.

80. The method of any one of paragraphs 73 to 79, wherein the first concentration of the test filter aid ranges from about 100 g/HL to about 600 g/HL.

81. The method of any one of paragraphs 73 to 80, wherein the first concentration of the test filter aid is equal to or greater than about 300 g/HL, for example equal to or greater than about 350 g/HL.

82. The method of any one of paragraphs 73 to 81, wherein the first concentration of the test filter aid is equal to or less than about 500 g/HL, for example equal to or less than about 450 g/HL.

83. The method of any one of paragraphs 73 to 82, wherein the feed material is a precursor in wine or beer production.

84. The method of any one of paragraphs 73 to 83, wherein the specified time period for measuring the volume of filtrate produced is equal to or less than about 60 seconds, for example equal to or less than about 30 seconds, for example equal to or less than about 15 seconds.

85. The method of any one of paragraphs 73 to 84, wherein the filtering of the test samples of the feed material and/or plurality of calibration samples of a calibration material with a first concentration of a test filter aid occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

86. The method of any one of paragraphs 73 to 85, wherein the calibration material comprises organic solids, for example compressible organic solids.

87. The method of any one of paragraphs 73 to 86, wherein the calibration material comprises barley malt extract.

88. The method of any one of paragraphs 76 to 87, wherein the desired pressure rate rise ranges from about 2 bar/hour to about 2.5 bar/hour.

89. The method of any one of paragraphs 76 to 88, wherein the specified time period for measuring the pressure rate rise ranges from about 1 minute to about 2 hours, for example from about 5 minutes to about 1 hour.

90. The method of any one of paragraphs 73 to 89, wherein the filter aid that is used to filter the filtrate of feed material produced by step a) or the filtrate of each calibration sample produced by step i) is a new sample.

91. A method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:

i) filtering a plurality of calibration samples of a calibration material with a first concentration of a test filter aid, wherein the calibration samples each have a different concentration of calibration material;

ii) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;

iii) filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid;

iv) measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample;

v) calculating the difference in volume of filtrate produced or time taken measured in steps ii) and iv);

vi) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples; and

vii) defining the relationship between the preferred concentration of filter aid determined in step vi) against the difference in volume of filtrate produced or time taken calculated in step v).

92. The method of paragraph 91, wherein the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of filter aid used;

C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

93. The method of paragraph 91 or 92, wherein the filter aid is a mineral filter aid.

94. The method of paragraph 93, wherein the mineral filter aid is diatomaceous earth and/or perlite.

95. The method of any one of paragraphs 91 to 94, wherein the filter aid is a low permeability filter aid, for example having a permeability between 0 and about 2 Darcy.

96. The method of any one of paragraphs 91 to 95, wherein the first concentration of the test filter aid ranges from about 100 g/HL to about 600 g/HL.

97. The method of any one of paragraphs 91 to 96, wherein the first concentration of the test filter aid is equal to or greater than about 300 g/HL, for example equal to or greater than about 350 g/HL.

98. The method of any one of paragraphs 91 to 97, wherein the first concentration of the test filter aid is equal to or less than about 500 g/HL, for example equal to or less than about 450 g/HL.

99. The method of any one of paragraphs 91 to 98, wherein the feed material is a precursor in wine or beer production.

100. The method of any one of paragraphs 91 to 99, wherein the specified time period for measuring the volume of filtrate produced is equal to or less than about 60 seconds, for example equal to or less than about 30 seconds, for example equal to or less than about 15 seconds.

101. The method of any one of paragraphs 91 to 100, wherein the filtering of the plurality of calibration samples of a calibration material with a first concentration of a test filter aid occurs at a pressure equal to or less than about 1 bar, for example equal to or less than about 0.6 bar, for example equal to or less than about 0.2 bar.

102. The method of any one of paragraphs 91 to 101, wherein the calibration material comprises organic solids, for example compressible organic solids.

103. The method of any one of paragraphs 91 to 102, wherein the calibration material comprises barley malt extract.

104. The method of any one of paragraphs 92 to 103, wherein the desired rate of pressure rise ranges from about 2 bar/hour to about 2.5 bar/hour.

105. The method of any one of paragraphs 92 to 104, wherein the specified time period for measuring the pressure rate rise ranges from about 1 minute to about 2 hours, for example from about 5 minutes to about 1 hour.

106. The method of any one of paragraphs 91 to 105, wherein the test filter aid used to filter the filtrate of each calibration sample produced in step i) is a new sample.

107. A method comprising:

a) filtering a first test sample of a feed material with a first concentration of a test filter aid;
b) filtering the filtrate produced in step a) with the same concentration of the test filter aid;
c) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced from the filtration of step b);
d) calculating the difference in volume of filtrate produced in the specified time period or the difference in the time taken for the specified amount of filtrate to be produced for the first test sample of the feed material (as measured in step c)) and water when filtered using the same concentration of the test filter aid.

108. The method of paragraph 107, wherein the method further comprises:

filtering a sample of water with the same concentration of the test filter aid; and

measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced.

109. The method of any one of paragraphs 107 to 108, wherein the wherein the filter aid is a mineral filter aid.

110. The method of any one of paragraphs 107 to 109, wherein the mineral filter aid is diatomaceous earth and/or perlite.

111. The method of any one of paragraphs 107 to 110, wherein the filter aid is a low permeability filter aid, for example having a permeability between 0 and about 2 Darcy.

112. The method of any one of paragraphs 107 to 111, wherein the first concentration of the test filter aid ranges from about 100 g/HL to about 600 g/HL.

113. The method of any one of paragraphs 107 to 112, wherein the first concentration of the test filter aid is equal to or greater than about 300 g/HL, for example equal to or greater than about 350 g/HL.

114. The method of any one of paragraphs 107 to 113, wherein the first concentration of the test filter aid is equal to or less than about 500 g/HL, for example equal to or less than about 450 g/HL.

115. The method of any one of paragraphs 107 to 114, wherein the feed material is a precursor in wine or beer production.

116. The method of any one of paragraphs 107 to 115, wherein the specified time period for measuring the volume of filtrate produced is equal to or less than about 60 seconds, for example equal to or less than about 30 seconds, for example equal to or less than about 15 seconds.

117. The method of any one of paragraphs 107 to 116, wherein the method is for predicting the ease of filtration of the feed material, wherein the value calculated in step d) decreases with increasing ease of filtration.

118. The method of paragraph 117, wherein increasing ease of filtration is increased speed of filtration of the feed material and/or decreased rate of pressure increase.

119. The method of any one of paragraphs 107 to 118, wherein the method is for designing a process for manufacturing a product, wherein the process for manufacturing comprises filtering the feed material.

120. The method of any one of paragraphs 107 to 119, wherein the method is for comparing the ease of filtration of one or more different feed materials, wherein the method is performed using a test sample of each feed material and feed materials with lower values calculated in step d) are easier to filter than feed materials with higher values calculated in step d).

## CLAIMS

1. A method for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising:
   a) filtering a first test sample of the feed material with a first concentration of a test filter aid;
   b) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced;
   c) using a calibration curve to select the concentration of filter aid to use based on the volume of filtrate produced or time taken measured in step b).

2. The method of claim 1, wherein the method further comprises, after step b):
   filtering the filtrate produced by step a) with the same concentration of the test filter aid,
   measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced, and
   calculating the difference in volume of filtrate produced or time taken for the first test sample of the feed material and the filtrate produced by step a),
   wherein step c) comprises using a calibration curve to select the concentration of filter aid to use based on the calculated difference in volume of filtrate produced or time taken.

3. The method of claim 1 or 2, wherein the calibration curve was obtained by a method comprising:
   i) filtering a plurality of calibration samples of a calibration material with the first concentration of the test filter aid, wherein the calibration samples each have a different concentration of calibration material;
   ii) measuring the volume of filtrate produced in the specified time period or measuring the time taken for a specified amount of filtrate to be produced for each calibration sample;
   iii) determining a preferred concentration of filter aid to use for filtration of each of the calibration samples;

iv) defining the relationship between the preferred concentration of filter aid determined in step iii) against the volume of filtrate produced in the specified time period or the time taken to filter the specified volume of filtrate measured in step ii).

4. The method of claim 3, wherein the calibration curve was obtained by a method further comprising, after step ii):

filtering the filtrate of each calibration sample produced by step i) with the same concentration of the test filter aid,

measuring the volume of filtrate produced in the specified time period or measuring the time taken for the specified amount of filtrate to be produced for each calibration sample, and

calculating the difference in volume of filtrate produced or time taken for each calibration sample of the feed material and the filtrate of each of the respective calibration samples produced by step i),

wherein step iv) comprises defining the relationship between the preferred concentration of filter aid determined in step iii) against the calculated difference in volume of filtrate produced or time taken.

5. The method of claim 3 or 4, wherein the preferred concentration of filter aid to use for filtration of a calibration sample is determined by a method comprising:

A) filtering the calibration sample using a plurality of concentrations of the test filter aid;

B) measuring the rate of pressure rise over a specified time period for each concentration of test filter aid used;

C) determining the minimum concentration of the test filter aid that provides a desired rate of pressure rise;

wherein the minimum concentration of the test filter aid that provides the desired pressure rate rise is the preferred concentration of filter aid to use for filtration of the calibration sample.

6. The method of any one of claims 1 to 5, wherein the calibration material comprises organic solids, for example compressible organic solids, for example wherein the calibration material comprises barley malt extract.

7. A method for creating a calibration curve for selecting the concentration of filter aid to use for the filtration of a feed material, the method comprising the steps of any one of claims 3 to 6.

8. A method for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising:
   a. filtering a first test sample of the feed material with a first filter aid to produce a first filtrate;
   a. measuring the turbidity of the first filtrate;
   b. filtering a second test sample of the feed material with a second filter aid having a different permeability to the first filter aid material to produce a second filtrate;
   c. measuring the turbidity of the second filtrate;
   d. defining the relationship between the permeability of filter aid and turbidity of the filtrate produced;
   e. selecting a desired turbidity of the feed material after filtration and using the calibration curve to select the permeability of filter aid to use based on the desired turbidity of the feed material after filtration.

9. The method of claim 8, wherein the method further comprises:
   filtering one or more further test samples of the feed material with one or more further filter aids having different permeabilities to the first and second filter aids to produce one or more further filtrates;
   measuring the turbidity of the one or more further filtrates;
   using the turbidity of the one or more further filtrate(s) to define the relationship between the permeability of filter aid and turbidity of the filtrate produced.

10. The method of any one of claims 1 to 9, wherein the filter aid is a mineral filter aid, for example wherein the mineral filter aid is diatomaceous earth and/or perlite.

11. The method of any one of claims 8 to 10, wherein the first filter aid has a permeability ranging from about 0.01 to about 1 darcy, for example from about 0.1 to about 0.5 darcy, and/or wherein the second filter aid has a permeability

ranging from about 3 to about 5 darcy, for example from about 3.5 to about 4.5 darcy.

12. The method of any one of claims 8 to 11, wherein the permeability of the filter aid having the highest permeability differs from the permeability of the filter aid having the lowest permeability by at least about 2 darcy, for example at least about 3 darcy.

13. The method of any one of claims 8 to 12, wherein the turbidity of the filtrate is measured after a certain volume has been collected or after a certain period of time.

14. A method for creating a calibration curve for selecting the permeability of filter aid to use for the filtration of a feed material, the method comprising the steps of any one of claims 8 to 13.

15. A method comprising:

a) filtering a first test sample of a feed material with a first concentration of a test filter aid;
b) filtering the filtrate produced in step a) with the same concentration of the test filter aid;
c) measuring the volume of filtrate produced in a specified time period or measuring the time taken for a specified amount of filtrate to be produced from the filtration of step b);
d) calculating the difference in volume of filtrate produced in the specified time period or the difference in the time taken for the specified amount of filtrate to be produced for the first test sample of the feed material (measured in step c)) and water when filtered using the same concentration of the test filter aid.

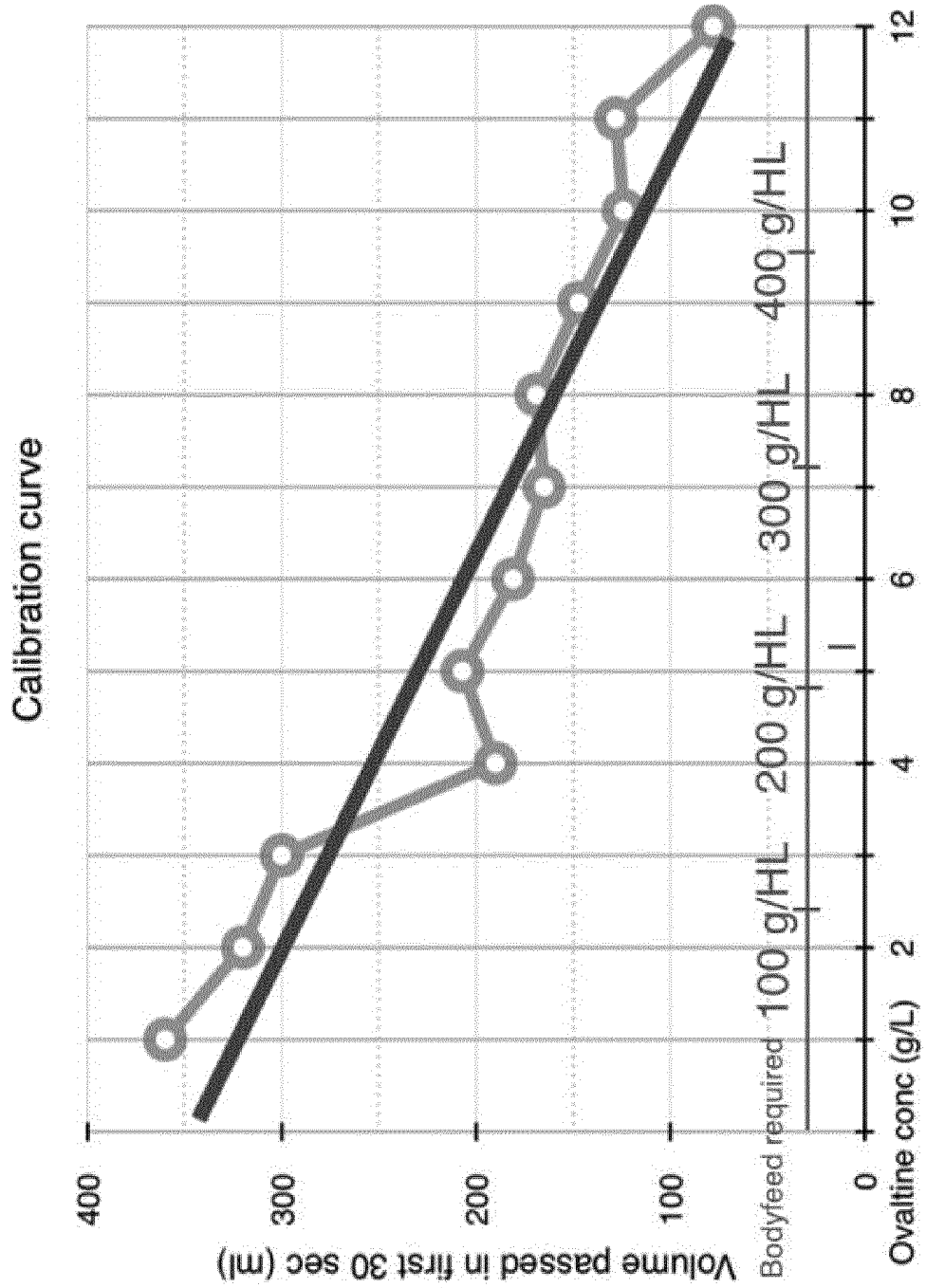
Calibration curve
Volume passed in first 30 sec (ml)
400
300
200
100
0
Ovaltine conc (g/L)
2
4
6
8
10
12
Bodyfeed required
100 g/HL
200 g/HL
300 g/HL
400 g/HL

FIG. 1

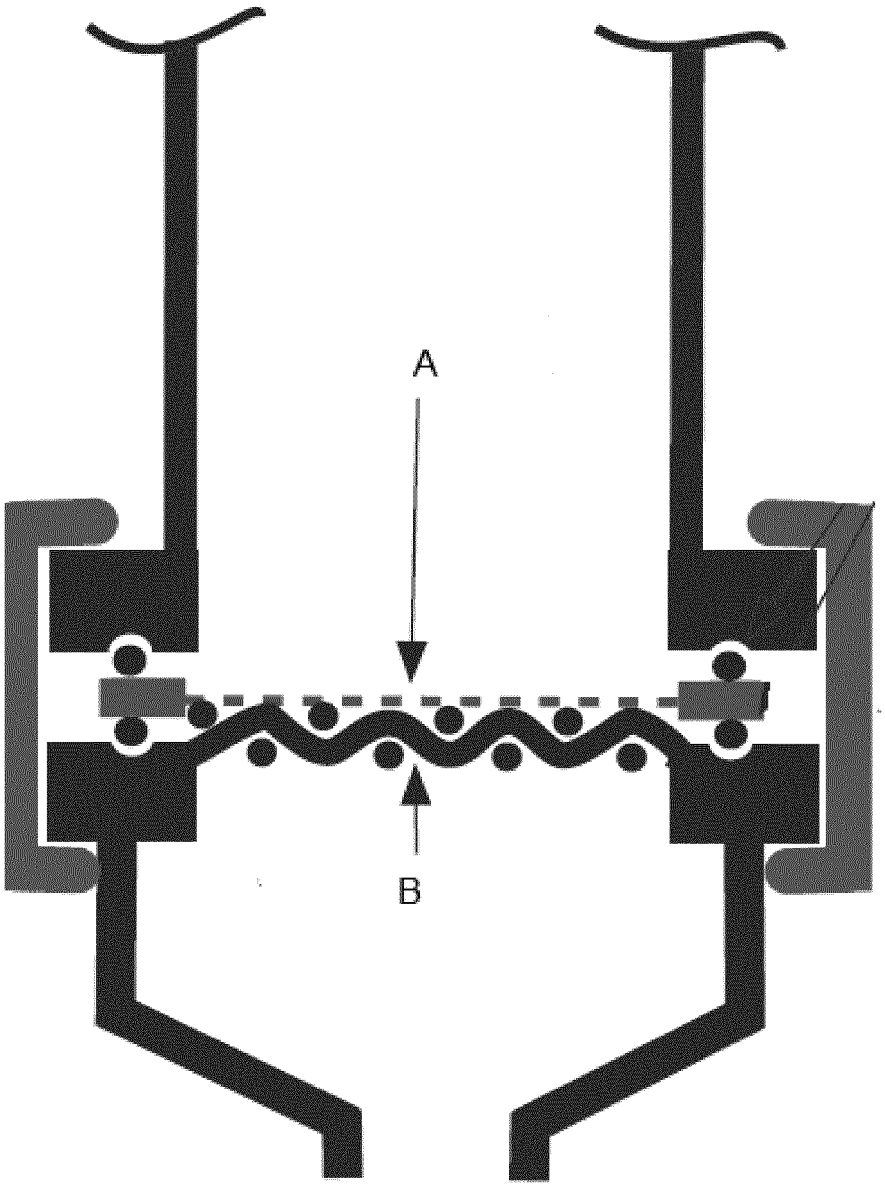


FIG. 2

# INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2018/068368

A. CLASSIFICATION OF SUBJECT MATTER
INV. G01N15/08 B01D37/02
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)
G01N B01D

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<br>Y | US 2005/244943 A1 (LADISCH MICHAEL R [US] ET AL) 3 November 2005 (2005-11-03)<br>paragraphs [0153], [0156], [0149]<br>----- | 1,6,7,10<br>2-5 |
| A | EP 2 006 367 A1 (MONDO MINERALS B V [NL])<br>24 December 2008 (2008-12-24)<br>paragraphs [0124], [0128]; figure 6<br>----- | 1-7 |
| A | US 2002/099174 A1 (JOHNSTON ANNA [AU] ET AL) 25 July 2002 (2002-07-25)<br>paragraph [0079]<br>----- | 1-7 |
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☐ 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 | Date of mailing of the international search report |
|---|---|
| 4 October 2018 | 10/12/2018 |

| Name and mailing address of the ISA/ | Authorized officer |
|---|---|
| European Patent Office, P.B. 5818 Patentlaan 2<br>NL - 2280 HV Rijswijk<br>Tel. (+31-70) 340-2040,<br>Fax: (+31-70) 340-3016 | Lokajova, Jana |

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# INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP2018/068368

**Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)**

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

**Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1-7(completely); 10(partially)

**Remark on Protest**

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

**FURTHER INFORMATION CONTINUED FROM PCT/ISA/** 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-7(completely); 10(partially)

   how to select the concentration of filter aid

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2. claims: 8, 9, 11-14(completely); 10(partially)

   how to select the the permeability of filter aid

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3. claim: 15

   how to determine the degree to which the solutes present in a feed material affect its filtration

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2018/068368

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
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