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(54) Abstract Title
Continuous filtration method and apparatus

(57) The improved method and apparatus provides for the use of at least two filters being arranged in series and wherein each filter has different properties and therefore a different role in the filtration process. The employment of such a method provides a continuous filtration system for filtering fatty acid esters out of reduced strength spirit without the need to significantly chill the filtrate prior to filtration. Such a method and apparatus can preferably be employed as part of a distillation process, particularly for whisky.

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Figure 1. Graph of Ester Levels v.'s Temperature

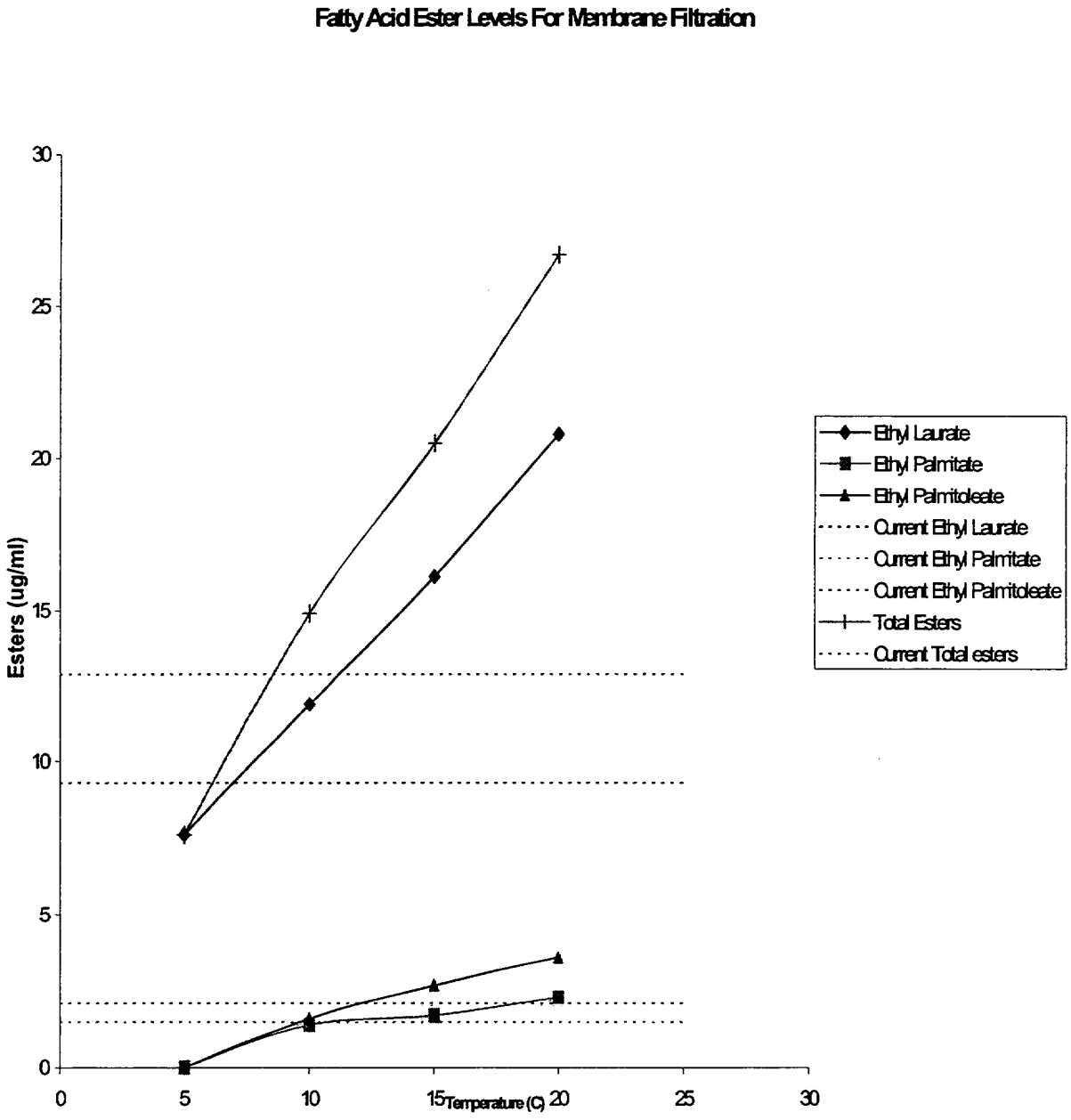
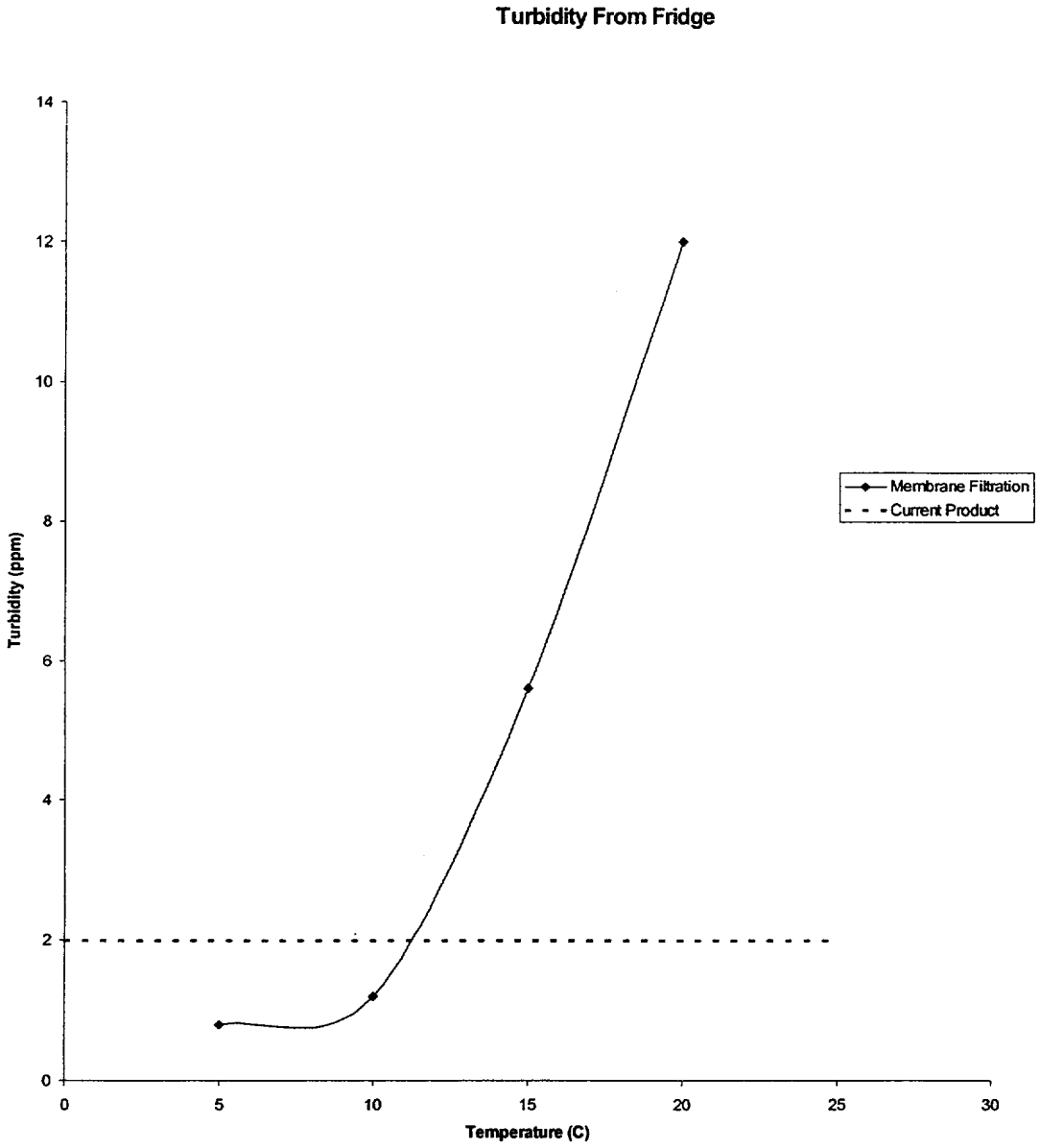


Figure 2. Graph of Turbidity v.'s Temperature



"Filter"

The present invention relates to an improved method and apparatus for the continuous filtration of specific substances during the alcohol distillation process.

Whisky and similarly produced alcoholic beverages are filtered in order to remove the haze which is formed when water is added to cask strength spirit to reduce it to bottling strength. When the water is added to the cask strength spirit the fatty acid esters come out of solution and make the whisky appear cloudy.

The whisky is chill filtered to ensure that the haze will not form in the bottle if the whisky temperature is reduced for example in a cold climate. Whisky filtration uses traditional technology. The general method uses a plate and frame filter press with cellulose acetate sheets. When the water has been added to the whisky the reduced spirit is left for at least

three hours and often longer before filtration, this being necessary for the filtration to be effective using this method. The whisky is then chill filtered between -10°C and +5°C depending on the production process employed by the whisky producer.

This chilling stage prior to filtration uses a lot of energy and is therefore very costly. The chilling also helps the fatty acid particles to agglomerate which makes it easier for the filters to remove them from solution. When starting the filtration, the whisky must be recycled for at least 15 minutes to ensure that particles of floc which come out of the filters are removed and do not enter the bottling vat. This recycling is also necessary to build up a primary filter cake on the filter. This is a layer of fatty acid ester particles which will in themselves act as filter and help to remove smaller particles which might otherwise get through the filter media.

This filtration process is a batch process as the rate of removal of the particles is not constant with time. The turbidity of the whisky is a measurement of how clear the product is, and is measured in ppm (parts per million). Throughout a batch the whisky turbidity will increase and by the end of the batch the turbidity of the whisky will be higher than the specification. However as this is a batch process, the turbidity in the batch will be within specification. This increase in turbidity is due to "breakthrough" from the filters.

Breakthrough occurs when the sheets become increasingly loaded with particles. It is then more difficult to force the whisky through the sheets causing an increase in the pressure drop across the filter sheets. As the whisky is forced through the sheets, some of the particles previously caught in the sheets breakthrough the filter causing the resultant turbidity increase.

When the filter sheets are exhausted they must be disposed of. Due to the nature of the sheets - typically approximately 1 litre of whisky per sheet is retained in the filter. This is lost product which must be disposed of together with the sheet since it cannot be recovered.

A change in the filtration technology was considered for whisky to find a more energy efficient system which can filter at a higher temperature. A filter which could be cleaned and therefore reused would also be desirable. A reduction in the loss of product in concomitant with filter replacement, would also be desirable, as would the replacement of existing batch processing techniques, with a filtration process which could be filtered immediately after the whisky had been reduced.

It is an object of the present invention to provide a continuous filtration system for filtering fatty acid esters out of reduced strength spirit without the need to significantly chill the filtrate prior to filtration.

According to the present invention there is provided a method of filtration which consists of a multi stage process with each stage containing a filter, with the filters being arranged in series.

Further, according to the present invention there is provided an apparatus for continuous filtration during a distillation process wherein the apparatus comprises at least two filters arranged in series wherein each filter has a different property and thus a different role in the filtration process.

In one particular embodiment the apparatus comprises three filters.

In an alternative embodiment of the invention, the invention provides an apparatus as described above which consists of only two filters arranged in series, wherein the two filters have different filtration properties.

Preferably the method of the invention will involve a three stage filtration process.

Preferably in such a three stage filtration process, each filter will be of a different type, with preferably the first filter of the series being a guard filter, a second filter being a prefilter and the third filter being a membrane filter.

Also preferably, there may be two filters in series, whereby the guard filter provided in the three filter

system is not present, due to it being located at an alternative stage in the filtration process. Preferably the final, or membrane filter, of the series will be composed of a material which is hydrophillic.

More preferably, the final membrane filter will be composed of polyvinylidene fluoride (PVDF).

Preferably the filtration will be carried out with the filtrate at a temperature of between 5°C and 25°C.

Most preferably, the filtration will be carried out of the filtrate at a temperature of between 8°C and 20°C.

Preferably filtration will be carried out at a defined pressure range, which will have been optimised to the filter size and type as used.

Preferred embodiments of the filtration device and process are described below.

The preferred embodiment of the present invention is a three filter train through which the whisky flows in series. Typically the invention consists of an initial guard (or nominal rating) filter followed by a pre-filter (combination of surface and depth) and a final membrane filter.

The system performance is sensitive to several variables: the type of filters to be used, the material used in the filter construction, the relative micron rating through the filter train, the upstream liquid

(whisky) temperature and the pressure drop across each filter.

The filtration media can vary for all three of the filters used. There are a large number of different membranes and filter media commercially available in cartridge form. Also the micron rating of the media could vary especially for the guard and the pre-filter and absolute rating filters could be used rather than nominal rating filters. If the whisky is left to sit after reduction, as is the case now a larger micron rating may also be possible for the final filter.

A two-filter system could be used rather than a three-filter system if there is a guard filter present elsewhere in the system. It is also possible that just a final membrane filter may be used although it is unlikely that this system would be successful for large batches.

It is also possible that membrane filter bags may be used instead of filter cartridges. The temperature used for the system could vary considerably as at the present time the temperatures used by different companies are different.

This technique can also benefit whisky production in other ways such as in the removal of sugary products thereby prolonging the lifetime of filters.

The filtration system consists of a heat exchanger to chill the whisky to the desired temperature and a

three-filter train. The filters are connected in series and the whisky flows through each in turn. The first filter housing contains guard filter cartridges these are 1 μm nominal rating filters. The purpose of these filters is to remove any large particles for example char from the casks. The second filter housing contains pre-filter cartridges and these are 0.2/0.45 μm nominal rating combined filters. The purpose of these filters is to remove larger particles and to protect the membrane filter as much as possible. The final filter housing contains 0.22 μm absolute rating membrane filter cartridges. These are the polishing filters to remove the small particles and will reduce the fatty acid ester levels to an acceptable level for bottling.

Suitable filters are known in the field. Examples of typical supplies include Millipore and Pall.

The temperature must be controlled closely as the temperature has a large impact on the quality of the final product.

There should be an upstream and a down stream pressure measurement for each of the filters. The pressure drop across each filter should be maintained below the set level. Excessive pressure will cause breakthrough from the filters. Maintaining the pressure drop causes the flow to fall below an acceptable level then this is a sign that the filters should be cleaned. It is possible that his could be used as a real time diagnosis of filter status.

The batch size through the filters will be dependent on the type of whisky or other similar product, which is being filtered and should be set for individual products. To prolong the lifetime of the filters, they should be cleaned before exhaustion as running heavily blocked filters causes excessive wear on the filters especially the membrane.

The invention is demonstrated with reference to the accompanying figures wherein;

Figure 1 illustrates fatty acid ester levels for membrane filtration, and

Figure 2 illustrates liquid turbidity as a function of temperature.

In whisky filtration, the pre-filtration (or upstream) whisky temperature is known to be a critical parameter. Tolerances of the order of ± 1-2°C have been shown to result in changes to system effectiveness as shown in Figure 1.

Studies have shown a performance sensitivity to choice of filter material. Specifically, the preferred choice of material is that which is hydrophilic. A typical material is polyvinylidene fluoride (PVDF). Alternatives such as cellulose acetate have been shown to work.

Filter train effectiveness is measured in a variety of ways as outlined in more detail below.

The liquid turbidity as a function of temperature has been measured and is shown in Figure 2. Other checks such as Arganoleptic testing, Chill haze stability, gas and liquid chromatography to respectively measure the ester and higher alcohol levels in the whisky are tests which have been undertaken and which quantify the improvements of the filter train.

Effective filtration using a two stage process has been shown on blended whisky. Also, using the two stage process, the whisky can be filtered immediately after reduction at ambient temperature - without any need to reduce the temperature of the whisky.

Experiment 1: 4" Filters

To determine the best membrane for the filtration and the temperature which the filtration process was to take place the filtration trials were carried out with 4" capsule filters. These filters are totally self contained with the pleated filter media inside a plastic capsule. The capsule has an inlet and outlet hose barb connection and vents to allow purging of air from the system. These filters therefore do not require a filter housing. The chilling was provided by a compressor which gave close temperature control to $\pm$ 1 °C. The 3 stage filtration process was set up by joining the 3 filters in series.

Three different types of membrane were tested, these being; the PVDF membrane from Millipore, the cellulose acetate membrane from Sartorius and the PES membrane

from PPD. In the initial 2" filter media disc trials, the Millipore membrane had performed best in the sensory analysis and so this was the membrane which was chosen initially to do the temperature trials on.

The aim of the trials was to create end products using membrane filtration, which were similar to the current products which are filtered using the traditional plate and frame filter press. This product also had to have chill haze stability. These success criteria are more vague than they appear due to the complexity of the products. The three main compounds which are responsible for chill haze instability are ethyl laurate, ethyl palmitate and ethyl palmitoleate. Some of the products were analysed for these esters during the trials.

Another way to check for chill haze instability is to put the products in the fridge and then check the turbidity, this was also carried out. There is an old method which used to be used by the Scotch Whisky Research Institute (SWRI) (a NAMAS accredited laboratory) to test for likelihood of chill haze instability called the Invergordon Chill Haze test and we also used this method. The problem with this method is that it is for blended whisky and so maximum levels are provided for blended whisky but not for Malt whisky. However we looked at several past bottling samples of 10 year old Malt whisky and took maximum values from that.

The first trials to be carried out were the temperature trials. The whisky was reduced and then filtered immediately at four different temperatures. These temperatures were 5°C, 10°C, 15°C and 20°C. Samples were taken during these trials and then the tests were carried out on these samples. The results can be found in table 1 and these are graphically represented in figure 1 and figure 2.

As can be seen from the results shown in figure 1, there is a direct correlation between ester removal and the temperature of filtration.

Figure 2 shows that there is also a correlation between temperature and turbidity. From these results the temperature was then chosen for the rest of the trials as 10°C.

More trials were then carried out with the 4" capsules to try to determine batch size. The results from these trials were erratic and difficult to explain. When the whisky is at full strength there is more ethyl palmitate present than ethyl palmitoleate, when the whisky is filtered this is reversed. In these sets of results there was more ethyl palmitate present than ethyl palmitoleate which would appear to indicate that the whisky had not in fact been filtered. This happened not only for the Millipore filters but also for the Sartorius filters which were also trialed at this point. The conditions were the same for all trials, however the end product was not the same. As this system was small it was not solid piped with stainless

steel pipework, braided plastic hose was used. This hose was connected at the time of the temperature trials. However for the next sets of trials the braided hose had been left with some alcohol in it and we believe that the hose reacted with the alcohol and these leachates contaminated the membranes and led to the filtration not being effective.

The contaminants may have partially blinded the membrane and this would have made filtration more difficult and caused an increase in pressure drop across the membrane and the particles may have been forced through. It was also difficult to control the pressure in this scale of experiment due to the size of the pump and the pressure was therefore higher than it should have been. This however confirms that control of the pressure in the final system will be very important. This trial provided data of what the whisky would be like if the filtration was not working. At this stage we also tried other membranes. The cellulose acetate membrane also appeared to work however the contamination also affected it. The Millipore membrane was selected due to the level of the technical support which the company provided, and also that the membrane had in the past produced whisky which had good sensory results. However other membranes would also have been an option and may be in the future.

Experiment 2: 10" Filter Cartridge Trials

Due to the problems with the smaller scale trials it was decided at this point to move to 10" cartridges

which provide five times the filtration surface area. These trials were carried out using existing solid pipework and an existing heat exchanger in the reduction area. The heat exchanger did not provide such close temperature control as the rented chiller, however, once the glycol was adjusted to the correct rate the temperature was constant. The trials also allowed a higher flow rate because of the surface area.

Experiment 3: Varying Whisky Type Used

1. 10 Year Old Malt Whisky

Trials at this stage were only carried out with Millipore filters, however different whiskies were filtered to see the effects of the filtration process. The three types chosen for the trials were a 10 year old Malt whisky, a blended whisky and Port Wood Finish whisky. The Port Wood Finish was chosen as to show that the new filters did not strip too much colour out of this whisky. These samples were analysed as before and also some sensory work was carried out on them.

The whisky was filtered first through the guard filter (Lifegard), then through the prefilter (Milligard) and finally through the membrane filter (Durapore). The batch size varied but was approximately 900 litres for the tests. 100 litres for this test was supposed to represent approximately 2000 litres on a full scale process. This was from calculations made at lab scale, however the flow rates from these filters would determine the end full scale filter size. Samples were

taken after each filter at 100 litre intervals and these were tested. The results from the 10 year old Malt trials are shown in table 2.
The turbidity of the samples was taken and recorded at room temperature, after being in the fridge for 2 days, at 3°C. The Invergordon chill haze stability value was also measured and these values are shown in table 3.

At all stages the results were compared to a current product sample and a judgement was made as to whether the membrane filters had produced a similar product to the current filtration method. From all the figures from this batch of trials it is likely that the chill haze stability would become a problem after 500 litres. Therefore the filters appear to be exhausted after 500 litres which would equate to a 10 000 litre batch size. This is smaller than was expected however the batch size would probably be larger than this in reality if the pressure was controlled more closely. However, if we are running a 2 filter system then this batch size would be fine as it would allow time for one filter train to be cleaned while the other was being used.

Sensory work was carried out on this product also and the difference was not found to be significant, the tests were repeated to ensure that the difference was not significant. The results from this sensory analysis are as follows.

Triangle Test 1

Number of correct responses 13

Number of incorrect responses 9

Number of correct responses 10
Number of incorrect responses 12

Triangle Test 2

Number of correct responses 4
Number of incorrect responses 18

Number of correct responses 9
Number of incorrect responses 13

The number of correct responses required for significance is 12 therefore the first time that test 1 was run it was significant, however the second time it was not significant. Test 1 was run on current product against the product taken from the membrane filtration process after 100 litres. This sample was taken when the temperature of the filtration process was too low and this may explain the difference as the ester levels in this sample were also low. The results from the taste test were as follows

Taste Test A

Number preferring current 10 Year Old Malt
3
Number preferring membrane filtered 10 Year Old Malt
1
Number with no preference
2

Taste Test B

Number preferring current 10 Year Old Malt

5

Number preferring membrane filtered 10 Year Old Malt

2

Number with no preference

3

2. Blended Whisky

The next stage was to try the filtration process with blended whisky. The whisky tested was H4325, this is a common blend. Similar tests were carried out however this product was not analysed for the long chain fatty acid esters as we had in the Malt whisky. The results from the analysis can be seen below in table 4.

Sensory work was also carried out on the blended whisky and the results can be found below.

Triangle Test

Number of correct responses	4
Number of incorrect responses	12

Again, the number of correct responses required for significance is 10 therefore the difference has been shown to be not significant. A taste test was also carried out on this and the results are shown below.

Taste Test

Number preferring current blended whisky

3

Number preferring membrane filtered blended whisky

2

Number with no preference

1

3. Port Wood Finish Whisky

Trials were then carried out on Port Wood Finish Whisky. As previously mentioned, the main reason for this was to ascertain that the new filtration process would not strip the pink blush from the whisky. These trials were also very successful in that the membrane filters successfully reduced the turbidity of the whisky to within acceptable levels and at the same time did not strip the pink blush from the whisky. The chill haze stability can be seen in table 5 however because the trials were carried out from a single cask, no bottling samples were available and therefore no sensory comparison could be carried out.

Also as the colour was important we measure the Dr Lange colour and the Corning colour. The Corning colour is measured at 430 nm and 490nm and these two figures are then subtracted. There is a specification for each wavelength as well as the difference and these are shown in table 6.

The results from the filtration trials are shown in table 7.

The colour difference is within specification however the actual colour measurements are above specification. This is because the casks was above specification and this would be blended away. However the results show that the filtration process is not stripping the colour out of the port wood finish whisky.

Sizing of Filter Train

For the sizing of the final filters the flow rate and the upstream pressure of the filters was measured. The upstream pressure gives a good indication of how much resistance is being caused to flow by the membrane. As the whisky is filtered the membrane becomes more loaded with the esters and therefore it will take more pressure to force the whisky through the filter. The best measure of how clean the membrane is, is the pressure drop across the filter. This is the difference between the upstream and downstream pressure of the filter and this will measure how difficult it is to get the whisky through the membrane. This will be an important measure on the larger scale as when the pressure drop gets above a certain level there is the possibility of break through on the filter. At this point the esters which have been removed may be forced into and through the membrane. This will obviously affect the turbidity and the taste of the whisky. The point at which break through occurs will help to determine the batch size of the filters. This will be estimated from the Invergordon chill haze turbidities and from ester levels. During the trials the pressure and the flow rate of the whisky was measured at

intervals and these results can be seen in table 8 below.

As can be seen from the results the pressure steadily increases as the loading on the membrane increases. The area of pressure drop across the filter is an important one for this process and there must be trade off between flow rate and upstream pressure. Further research will show what our maximum process pressure should be and what the preferred operating pressure will be. This will be different for each of the filters and should be decided at the pilot plant scale of the filtration process. It may be possible to keep the pressure drop more constant by introducing a back pulsing system which will send a pulse of air through the membrane when the pressure drop reaches a certain level and this may clean off some of the surface debris and allow the whisky to flow more freely.

The filter train was sized according to the flow rate which is required and the flow rates which we achieved through one 10" filter. The flow rate taken is not the flow rate at the start of the run, it is the flow rate which we can achieve at the end of a run. It is assumed that the filter can be cleaned to at least 80% of it's original performance and the sizing is based on this.

The housings are all oversized as 10" and 20" cartridges can both also be used in the 30" filter housing to reduce cost and surface area and also some of the inlets can be blanked off to reduce the surface area. This sizing gives greater flexibility. Also the

housings come in certain standard sizes and it is more cost effective to buy the standard sizes than to get specific sizes made to order.

Cleaning Of Filters

The cleaning of the filter cartridges was the next challenge, as, if the filters cannot be cleaned up properly then they may only be used once and not changed less frequently. The first method of cleaning which was tried was hot water, both forward and backward flush. However the esters are not water soluble and this did not work. After consulting a textbook and the SWRI for help in this matter it was decided that there were two possibilities. One was detergent and the other was neutral spirit. Although there was a limited amount of neutral spirit available there was not enough to flush the filters, therefore pieces of the membrane cut from an exhausted 4" capsule were soaked in four different solutions, these were:

1. Hot water
2. Hot water and detergent
3. 50% neutral spirit
4. 96% neutral spirit

The membranes were soaked overnight and then removed from the solutions. After inspecting all the samples with a scanning electron microscope (SEM) it could be seen that all except the hot water cleaned the filters.

Next the 10" cartridges were soaked in the neutral spirit overnight, and this did remove some of the esters however for effective cleaning a dynamic flush is required. Using neutral spirit will create a number of other problems such as accounting to Customs & Excise for the spirit and if 96% spirit is to be used then the area will need to be a zone 1 area and this increases equipment and instrumentation costs and will cut down on the choice of sensors which we can use. Also there is the added cost of disposal and the neutral spirit will require to be cleaned up if it is to be reused. It therefore does not seem to be a very practical solution.

The best solution would appear to be detergent and warm water, with a final rinse with demin water. A company called Henkel have been contacted about possible suitable detergents from their range and their membrane cleaning expert is presently looking into this. The detergent solution would then go to the foul drain on site. This would therefore appear to be the most cost effective and easiest option.

More trials will therefore be carried out on the 10" filter cartridges to try and clean the filters effectively. This is very important to ensure that an economically viable lifetime can be obtained from the filters. The filters should be able to be cleaned to obtain at least 80% of their original performance. The lifetime of the filters is also dependent on the filters not being run at extreme pressures and it is better to clean the filters more regularly as running

them when they are heavily loaded with filtrate causes stress on the filtration media.

Summary

1. The use of membrane filtration has a number of advantages over the plate and frame filter press: Firstly the whisky can be filtered at 6°C higher than currently. Secondly the cartridges are cleanable and therefore reusable and would have to be changed probably every 3-4 months instead of after every batch as is currently the case. The whisky can be filtered immediately after reduction using the cartridges and no recycling is necessary, as there is no shedding of floc from the filters. After use the cartridges can be air purged and this reduces the hold up volume and makes the losses in product considerably less than the current technology. The cartridges can be used as part of a continuous process. The process can be used in number of different drinks industries where removal of chill haze is a problem e.g. American, Canadian, Irish and Japanese whiskeys, in brandies in wines and in certain beers.

   One of the key benefits of the multistage filter is the fact that filtration can take place without the need to chill the whisky. The commercial benefit of the invention being seen in the reduced energy costs for the producer.

Table 1: Temperature Trial Analysis Results

	Current Product	Millipore @ 5C	Millipore @ 10C	Millipore @ 15C	Millipore @ 20C
Ethyl Laurate (ug/ml)	9.3	7.6	11.9	16.1	20.8
Ethyl Palmitate (ug/ml)	1.5	0	1.4	1.7	2.3
Ethyl Palmitoleate (ug/ml)	2.1	0	1.6	2.7	3.6
Total Esters (ug/ml)	12.9	7.6	14.9	20.5	26.7
Turbidity (room temp), ppm	0.6	0.4	0.35	0.55	0.65
Turbidity (from fridge) , ppm	2	0.8	1.2	5.6	12
Invergordon Turbidity, ppm	8.7	6.4	7.1	15.6	30.6

	Lifegard 100L	Lifegard 500 L	Lifegard 900 L	Milligard 100 L	Milligard 500 L	Milligard 900 L
Ethyl Laurate (ug/ml)	16.8	18.7	23.3	16.1	15.2	17.6
Ethyl Palmitate (ug/ml)	13	8.6	17.8	1.7	4.3	7.9
Ethyl Palmitoleate (ug/ml)	10.7	7.3	13.7	1.9	4.5	6.7
Total Esters (ug/ml)	40.5	34.6	54.8	19.7	24	32.2
Turbidity (room temp), ppm	12.8	0.75	0.95	0.25	0.35	0.55
Invergordon Turbidity, ppm	58	36	38	16.6	36	31.5

Table 2a. Analysis results: 10 Year Old Malt with 10" Cartridge Trials.

	Current Product	Durapore 100	Durapore 300	Durapore 600	Durapore 900
Ethyl Laurate (ug/ml)	7.6	12.9	14.1	14.3	13.9
Ethyl Palmitate (ug/ml)	1.5	1.7	1.6	3	2.5
Ethyl Palmitoleate (ug/ml)	1.5	2.3	2.3	3.2	3.1
Total Esters (ug/ml)	10.6	16.9	18	20.5	19.5
Turbidity (room temp), ppm	0.45	0.35	0.2	0.25	0.3
Invergordon Turbidity, ppm	7.4	8.9	9.8	17.4	19.4

Table 2b. Analysis results: 10 Year Old Malt with 10" Cartridge Trials.

	Turbidity at Room temp	Turbidity in Fridge 2 days	Turbidity at 3 °C	Invergordon Chill Haze
Durapore 100L	0.55	0.55	2.3	8.9
Durapore 200L	0.6	0.6	2.3	4.4
Durapore 300L	0.65	0.65	2.95	9.8
Durapore 400L	0.75	0.75	3.1	9.2
Durapore 500L	0.85	0.85	3.4	15.8
Durapore 600L	1.6	1.6	4.8	17.4
Durapore 700L	1.7	1.7	5.2	17.4
Durapore 800L	1.45	1.95	6.2	19.4
Current Product	0.7	0.7	2.85	7.4

Table 3. 10 Year Old Malt Chill Haze Stability Results

	Turbidity at Room temp	Turbidity at 3oC	Invergordon Chill Haze
Durapore 100L	0.8	2.5	4.4
Durapore 200L	0.8	2	3.8
Durapore 300L	0.8	2.2	3.4
Durapore 400L	0.8	2.6	4
Durapore 500L	0.8	2.35	4.15
Durapore 600L	0.8	2.4	4.1
Durapore 700L	1.15	2.75	3.8
Durapore 800L	1.15	1.8	3.55
Current Product	0.95	2.4	3.65

Table 4. Blended Whisky Chill Haze Stability Results

	Turbidity at Room temp	Invergordon Chill Haze	Turbidity in Fridge 2 days
Durapore 100L	0.75	46	5.8
Durapore 200L	0.7	32.5	2.35
Durapore 300L	0.75	37.5	2.8
Durapore 400L	0.8	40.5	2.8
Durapore 500L	0.75	42.5	2.45
Durapore 600L	0.7	38.5	2.35
Durapore 650L	0.7	44	2.1
Current Product	1.05	64	13.4

Table 5. Chill Haze Stability Results for Port Wood Finish Whisky

	Minimum	Target	Maximum
Dr Lange	12	12.5	13.5
430nm	0.2	0.23	0.26
490nm	0.23	0.25	0.27
430-490	-0.04	-0.02	0.03

Table 6. Colour Specifications for Port Wood Finish Whisky

Litres	430nm	490nm	430 - 490	Dr Lange
100	29	29	0	12.9
200	30	30	0	13
300	28	28	0	13
400	30	30	0	13
500	29	28	0.01	13
600	30	31	-0.01	13
650	26	27	-0.01	13

Table 7. Colour Measurements For PWF From Membrane Filtration Trials

10 Year Old Malt

	Pressure (bar)		
Litres	**Lifegard**	**Milligard**	**Durapore**
100	0.1	0.5	0.8
200	0.1	0.6	1
300	0.1	0.7	1.2
400	0.3	0.75	1.25
500	0.4	0.8	1.25
600	0.5	0.8	1.4
700	0.5	0.8	1.4
800	0.55	0.8	1.5
900	0.6	0.85	1.5

Table 8a. Upstream Pressure of Filters, 10" Trials.

Blended

Whisky

	Pressure (bar)		
Litres	**Lifegard**	**Milligard**	**Durapore**
100		0.8	0.8
200	0.4	0.85	0.9
300		0.8	0.95
400	0.45	0.7	1
500		0.85	1.05
600	0.6	1	1.1
700		0.9	1.2
800	0.8	0.95	1.2

Table 8b. Upstream Pressure of Filters, 10" Trials.

Port Wood

Finish

	Pressure (bar)		
Litres	**Lifegard**	**Milligard**	**Durapore**
100	0.1	1.2	0.8
200	0.1	1.2	1
300	0.2	1	1.1
400	0.4	0.6	1.1
500	0.4	1	1.1
600	0.5	1	1.15
650	0.5		1.15

Table 8c. Upstream Pressure of Filters, 10" Trials.

Table 9: Blended Whisky

Flow rate required 120 l/min

	Guard Filter	**Prefilter**	**Membrane Filter**
Flow Rate (10")	30 l/min	10 l/min	10 l/min
Filter Size	6 x 10"	5 x 30"	5 x 30"
List Price of Filters	£270	£1200	£1845
Housing Size	3 x 30"	7 x 30"	7 x 30"
Housing Price	£2000	£4000	£4000

Table 10: Malt Whisky

Flow rate required 150 l/min

	Guard Filter	**Prefilter**	**Membrane Filter**
Flow Rate (10")	20 l/min	8 l/min	7 l/min
Filter Size	4 x 30"	8 x 30"	8 x 30"
List Price of Filters	£540	£1920	£2952
Housing Size	5 x 30"	12 x 30"	12 x 30"
Housing Price	£3000	£7000	£7000

CLAIMS

1. A continuous filtration method for filtering fatty acid esters out of reduced strength spirit without the need to significantly chill the filtrate prior to filtration, the method consisting essentially of a multi stage process with each stage containing a filter, with the filters being arranged in series.

2. An apparatus for continuous filtration during a distillation process wherein the apparatus consists essentially of at least two filters arranged in series wherein each filter has a different property and thus a different role in the filtration process.

3. An apparatus as claimed in claim 2 which consists essentially of only three filters arranged in series, wherein the three filters have different filtration properties.

4. A method as claimed in claim 1 wherein the multi state process is a three stage filtration process.

5. An apparatus as claimed in claim 3 wherein each filter will be of a different type, with the first filter of the series being a guard filter, the second filter being a prefilter and the third filter being a membrane filter.

6. An apparatus as claimed in claims 2, 3 or 5 wherein the final, or membrane filter, of the series is composed of a material which is hydrophillic.

7. An apparatus as claimed in claims 2, 3, 5 or 6 wherein the final membrane filter is composed of polyvinylidene fluoride (PVDF).

8. A method as claimed in claim 1 or 4 wherein the filtration is carried out with the filtrate at a temperature of between 5°C and 25°C.

Application No: GB 0021632.5 **Examiner:** L.V.Thomas
Claims searched: 1-8 **Date of search:** 28 February 2001

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): C6E (EJCB, EJCD); B1D (DNUD, DQAA, DQAX)

Int Cl (Ed.7): C12H 1/07, 1/16; B01D 36/02

Other: Online: EPODOC, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 1045191	(MILLIPORE) see p.1 ll.81-88 and p.2 ll.8-37	2,6
X	EP 0691151 A2	(ME-COM) see p.2 ll.22-30 and p.2 l.44 - p.3 l.16	2-4
X	EP 0464322 A1	(PERDOMINI) see col.1 ll.1-31, col.3 ll.14-23 and col.6 ll.3-7	2-4,6,7
A	EP 0208450 A2	(APV INT.) see p.3 ll.9-24, p.5 ll.13-25 and p.6 ll.7-25	

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.