

IMPROVEMENTS IN AND RELATING TO THE PROCESSING OF MATERIALS
This invention relates to the extraction of useful com-

pounds from material of vegetable origin and has been devised particularly through not solely to provide an improved extraction method and apparatus for the recovery of these useful

5. compounds. Thus, the invention relates to liquid-solid extraction, the solid being any material of vegetable origin.

- For many years useful compounds have been extracted from material of vegetable origin, the compounds being used in beverages adhesives and medicines for example. The general
10. principal which has been commonly used to extract these useful compounds is to contact a suitable solvent and the vegetable material and after a prescribed time, to separate this mixture into a solute-laden solvent phase containing the useful compound or compounds and a substantially solid phase, being the spent
15. vegetable material from which some of the compounds have been removed. The useful compounds are often concentrated or removed altogether from the solvent phase by evaporation such as spray drying and the spent vegetable material is either discarded, or as described and claimed in New Zealand patent
20. specification No. 184076, possibly used as a base for the preparation of plant growing mediums.

- Typically, prior art liquid-solid extraction of the type to which this invention relates involves a multi-stage counter current system based on either immersion or percolation,
25. wherein some degree of separation between liquid and solid phases takes place between stages so that the solid phase can be passed in the opposite or counter current direction to the

liquid phase.

An advantage of the counter current extraction system is that the solid vegetable material after passing through the system is washed by new solvent entering the system so

5. that little of the useful extract is lost, the same solvent becoming richer in useful compound content or solute until it finally emerges at greatest strength at the vessel or cell where new solid vegetable material is introduced. Presses or centrifugal phase separators are sometimes used to separate

10. the solid phase and the liquid phase, but by far the most common method employed is to arrange vessels or cells so that the liquid phase can percolate through the solid vegetable material in each, to be introduced at the top of the next vessel or cell to again percolate through another bed of the

15. vegetable material. Usually, although not essentially, the vessels or cells move so that eventually each reaches a station on the apparatus where spent vegetable material is discarded and the empty vessel or cell is filled with new vegetable material to again repeat a cyclic path through the

20. system. For a given size of apparatus production is limited by the rate of percolation of the solvent through the bed of vegetable material which in turn is in conflict with efficient recovery of useful compounds from a given quantity of vegetable material, because the vegetable material has to be comminuted,

25. with finer particles providing the greatest yields as shown for example, in Figure 2 of New Zealand patent specification No. 179933, but finer particles also giving lesser percolation rates. A compromise has to be reached where percolation rate

and yield are balanced to the extent that an adequate production rate can be maintained while a proportion of the useful compound is often discarded, being still contained in the spent vegetable material. Since the several vessels or cells used are serially
5. connected to use the counter current extraction system the flow of solvent through each and every vessel is governed by the percolation rate and therefore the rate of filling with compound containing solvent, of the vessel or cell which has just been charged with new vegetable material is also governed by the
10. percolation rate of the vegetable material beds in each vessel or cell and the process is again limited by the percolation rate.

New Zealand patent specification No. 181512 discloses a method whereby productivity can be increased by filling
15. extraction vessels with compound containing solvent as well as new vegetable material before introducing them to the extraction line with the objective of reducing the limitations imposed by the percolation rate, and while this constitutes an improvement in the operation of the prior art apparatus to which
20. New Zealand patent No. 181512 relates, operation of the apparatus is still limited by the percolation rate of solvent through the beds of vegetable material.

An example of a typical multi-stage liquid-solid extractor employing a plurality of vessels or cells is the Rotocell,
25. there being a number of apparatus using the same multi-stage counter current principle, some of which have moving vessels or cells, and others of which have stationary vessels or cells with a phase separation means therebetween.

Closely related to the Rotocell is the prior art endless belt extractor which uses the same principle of operation, the percolation rate determining the belt speed and amount of drainage area required, which in turn limits the

5. minimum mean particle size which can be accepted. An example of a typical endless belt extractor is the De Smet extractor wherein a solids feed forms a bed on the belt, the bed being subsequently sprayed with solvent liquid which percolates through the bed to accomplish extraction. In
10. common with the Rotocell, the De Smet extractor employs a bed which is typically 1 metre or more in depth, and it can be seen that a low percolation rate would require the use of prohibitively large apparatus to enable sufficient drainage area to be provided.
15. The conflict between percolation rate and yield still remains however, and full advantage cannot be taken at present of the fact that greater yields can be obtained when the vegetable material exists as smaller particles.

There is a need for an improved method and apparatus

20. suitable for extracting useful compounds from finely divided vegetable material.

It is an object of ~~one aspect~~ of this invention to provide a method ~~and/or apparatus~~ for extracting useful compounds from vegetable material.

25. It is a further object of ~~one aspect~~ of this invention to provide a method ~~and/or apparatus~~ for extracting useful compounds from vegetable material, which overcomes or reduces some of the difficulties experienced with prior art methods, or which at least provides the public with a

useful choice.

Other objects and/or advantages of various forms of the invention, will become apparent from the following description.

5 According to one aspect of this invention, there is provided a method of extracting useful compounds from vegetable matter, (as herein defined), said method including the steps of:

- 10 (1) preparing a mixture of the vegetable matter and at least one solvent;
- (2) allowing said mixture to reside in at least one vessel for a substantially predetermined time;
- 15 (3) thereafter transferring the mixture from said at least one vessel to a screen so that a mat or cake is formed on said screen, whereby the solvent phase separates from the vegetable phase by passing through said screen;
- 20 (4) recovering the solvent phase and obtaining useful compounds extracted from the vegetable matter as a filtrate from said screen.

The term "vegetable matter" as herein used throughout the specification and claims, is herein defined as comprising particles of vegetable matter, of such a
25 size as to permit a solvent to pass through a mat or cake of said particles being one metre thick, at a rate of less than 50 litres per square decimeter per hour.

This invention which includes the foregoing will now be described by way of example with reference to the accompanying drawings, wherein:

- Figure 1: is a graph showing the relationship between vegetable material diffusivity and particle size.
- Figure 2: is a graph showing the relationship of coffee bean diffusivity and particle size.
- Figure 3: is a further graph showing extraction rate with tea using hot water solvent.
- Figure 4: is a diagrammatic drawing of one basic form of method and apparatus for the extraction and recovery of compounds from vegetable matter in accordance with this invention.
- Figure 5, is a detailed block diagrammatic view of a process and apparatus according to one form of the invention.
- Figure 6: is a graph showing the process using pinus radiata bark.

Many useful compounds are recovered from vegetable material using a process based upon percolation of a suitable solvent through particulate vegetable material, the useful compounds being extracted into the solvent phase to be further treated usually by evaporation to separate most or all of the solvent from the useful compounds. Often the solvent is water or water to which chemicals intended to aid extraction

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have been added or introduced, although other solvents are also used. Sometimes the solvents are recovered from a condensation stage during concentration by evaporation, to be re-used.

5. Examples only of vegetable materials from which useful compounds may be extracted according to this invention include leaf, bark, root, seed and blossom for example, the compounds being used in products such as beverages, pharmaceutical products, adhesives, spices, dyes and fragrances.

10. Extraction of coffee and tea for the manufacture of instant coffee and tea, and extraction of polyphenolic materials from bark for the manufacture of adhesives or tanning agents are particular examples. In these examples water may be used as a solvent, and in the extraction of

15. polyphenolic materials from bark we prefer to add suitable stabilizing compounds and/or suitable extractant compounds to the water as disclosed in New Zealand patent specification No. 179933, and as further described hereinafter. The water is typically used at elevated temperature.

20. In some examples such as coffee the vegetable material is roasted prior to extraction while in others it is preferred to execute the extraction while the vegetable material is in a substantially green condition, that is, while sap or natural moisture is still present, in the vegetable matter, and

25. vegetable material in either of these conditions or in some other condition can be processed using the method and apparatus of this invention.

While in prior art Carrousel extractors the yields depend to some extent on the number of extraction steps or stages the most significant single factor when extraction conditions are optimised becomes the particle size or degree

5. of refinement of the vegetable material. Our experience has shown that the minimum percolation rate through the bed which can be tolerated for economic reasons governs the particle size or degree of refinement which can be accepted by prior art methods and apparatus and that when the
10. minimum percolation rate is reached the particles are still too large (refining is insufficient) to take full advantage of the increased yields and increased extraction rates which can result from a greater degree of refining as disclosed in New Zealand patent No. 179933 (Figure 2 of the specification)
15. because such refining reduces the percolation rate to a generally unacceptable level. A related problem referred to in the prior art, as "fines plugging" is often experienced and it is commonly recognised that prior art apparatus require a feedstock of consistent mean particle size related to the
20. percolation rate, with the minimum of fine particles known to cause plugging or blockage. In some operations it has been found necessary to screen out the fine particles to avoid fines plugging which of course means that a proportion of the vegetable material feedstock is wastefully discarded.
25. Liquid-solid extraction as it relates to this invention depends mainly upon diffusional mechanisms whereby material is diffused from the interior of a relatively solid particle, and we have found that the rate of extraction depends considerably upon the extent to which barriers to diffusion have been broken

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down by mechanical means. Thus, the diffusional mechanisms which occur during liquid-solid extraction of vegetable material, especially where they relate to extraction rate and yield, and especially with respect to this invention can be

5. simply described in terms of the vegetable material diffusivity and the vegetable material thickness, that is, the particle size.

The effect of vegetable material diffusivity and particle size is clearly illustrated in Figures 1 and 2 herein.

10. To prepare data for Figure 1, *Pinus radiata* bark was comminuted after removal from a freshly felled tree. The comminuted bark was sieved to produce mean particle size fractions which were then each extracted with water at 90°C for a period of 30 minutes. It can be clearly seen from Figure
15. 1 that ignoring the practical problems of apparatus operation there is an advantage in reduced particle size so far as the yield and rate of extraction is concerned, and hence the economics of producing such extracts can be affected.

- Now considering the practical problems of prior art apparatus
20. operations however we find that whereas Figure 1 clearly shows that there are advantages in reduced particle size, the percolation rate falls well below 50 litres per square decimetre per hour before the advantage can be used. A percolation rate of 40 to 50 litres per square decimetre per hour is consid-
25. ered to be near the lower limit for prior art extractors of the Rotocell or the De Smet type for example. This invention enables percolation rates of less than 50 litres per square decimetre per 1 metre deep of bed, per hour to be used, 10 litres per square decimetre per hour for example

being perfectly satisfactory.

Now referring to Figure 2 herein a different aspect of the interaction between diffusivity and vegetable material particle size is shown. To prepare Figure 2, we have taken 5. commercial roasted coffee ground to an acceptable percolation rate for use in prior art extractors. A portion of this coffee was extracted for various periods of time (residence time) as received, while a second portion of the same coffee was deliberately ground to a fineness which had an unacceptably 10. low percolation rate with respect to operation of the prior art Carrousel type apparatus, and this second portion also was extracted for various periods of time. The results are shown in Figure 2.

In Figure 2 the results shown as CC refer to the roasted 15. and ground coffee as received while the results shown as FC refer to the second portion of coffee which was extracted after fine grinding. It must be appreciated that Figure 2 is a generalisation only since there are varieties of coffee and degrees of roasting. Nevertheless, Figure 2 clearly 20. shows that for a vegetable material of given diffusivity - the particular coffee sample, diffusional mechanisms depend upon the particle size which of course defines the length of the diffusion path. The particle size also determines the percolation rate which in turn defines the operational 25. constrains of prior art extractors. With reference to Figure 2, it can be seen that these factors are of considerable importance when the productivity and economics of operating

extraction methods and apparatus are considered as the yield of extract from the coarse coffee after extraction for 120 minutes is similar to that obtained after extraction of the fine coffee for only 10 minutes, and the yield from fine coffee after extraction for 60 minutes is greater than that of the coarse coffee after extraction for 120 minutes. It is not unusual for prior art extractors to require a residence time of 120 minutes. The present invention relates to liquid-solid extraction wherein the solid is any material of vegetable origin, whether such material is extracted while in a fresh or green condition, or wherein such material is extracted after suitable pretreatment which in the case of coffee for example constitutes roasting of the coffee beans. The diffusional mechanisms described herein apply to most vegetable material feedstocks which can be extracted according to this invention, although some feedstocks when extracted involve mechanisms which are partially diffusion and partially washing from the feedstock surface. An example of a vegetable material feedstock which when extracted involves mechanisms which are partially diffusional and partially washing is tea. Indeed, according to Kirk-Othmer, Encyclopedia of Chemical Technology Volume 9, page 735 the extraction of tea is predominantly washing, although we note that the diffusion paths are short as well, whether the tea leaf is fine ground or not. Our experiments with fine and coarse ground tea have produced the results shown in Figure 3 herein which show that there is little advantage in fine grinding tea leaf, probably because of the

relationship of the thin leaf with its short diffusion paths to our explanation of diffusional mechanisms and to the washing explanation given by Kirk-Othmer. Figure 3 shows that the extraction rate with tea using hot water solvent

5. is rapid whether fine or coarse tea is the feedstock, and is virtually complete after a residence time of about 10 minutes. Residence time is the term used in the art for the contact time between solvent and the vegetable material feedstock. It seems likely to us that the small discrepancy

10. in yield shown in Figure 3 between the finely ground tea (FT) and the tea leaf as received (CT) can be explained in terms of fines suspension in the FT solute as it has been found difficult to completely free the solute from suspended solids unless special filtration techniques are resorted to, to

15. remove all of the colloidal suspension therefrom.

Figure 3 clearly shows that diffusion is rapid when the diffusion path is short, and accordingly, the results support the advantage to be gained from using vegetable material feedstocks in the form of small particles, notwithstanding that

20. the advantage to be gained with tea is small. The advantage to be gained with bark or coffee for example is much greater than that to be gained with tea.

For the efficient performance of this invention and in particular to enable advantage to be taken of vegetable material

25. of relatively small particle size and the increased yield and/or increased rate of extraction resulting from the use thereof, apparatus according to an aspect of this invention is provided. Said apparatus according to an aspect of this invention consists in at least one vessel and a suitable

screen of an endless or continuous loop type associated therewith, examples of said suitable screen being a circular table or a disc type, a circular drum type and an endless belt type. Coacting with said suitable screen, a plurality of

5. receiving chambers are provided , said receiving chambers defining phase separation stations on the suitable screen. The versatile apparatus, by the connection of adequate piping may be operated with counter-current washing extraction and recovery zones while at the same time enabling controlled
10. contact between solvent and vegetable material for a pre-selected residence time, yet avoiding the serious prior art problems resulting from inadequate percolation rates.

- In operation of apparatus according to an aspect of this invention contact of suitable solvent and vegetable material
15. is initiated within said at least one vessel by the formation of a slurry which moves in substantially plug flow through a vessel to be discharged at an end thereof and deposited in the form of a mat cake or bed on the suitable screen which moves to transport the mat cake or bed causing it, by movement
 20. of the screen to pass in sequence over a plurality of phase separating stations defined by receiving chambers on the other side of the screen. After passing over all the phase separating stations, being transported by the screen movement, either intermittent or continuous, the mat cake or bed is discharged
 25. from the screen, being largely exhausted of useful compounds which are now contained in a solute or miscella recovered from a receiving chamber. Underflow, being largely solute free solvent remaining in the discharged mat cake or bed may be

recovered therefrom by pressing, to be returned to the extraction system if desired. The solute or miscella recovered from a receiving chamber may be concentrated or otherwise treated dependent upon the nature thereof and the uses for which it is intended.

The apparatus of this invention requires at least one vessel, in which contact between suitable solvent and vegetable material feedstock takes place to provide residence time during which diffusional mechanisms as described hereinbefore take place.

10. Apparatus can be operated according to this invention in which one vessel only is provided and said vessel may be of an open or closed type, or of a pressure type to enable contact between suitable solvent and vegetable material feedstock to include a treatment at above atmospheric pressure. Treatment at pressure,
15. or at least in a closed top vessel is an advantage for example when vegetable material feedstock contains aromatic substances of interest which can be recovered from a vapour phase drawn from a closed top vessel and subsequently condensed. Such a treatment can be used in coffee extraction for example, the
20. aromatic fraction being first recovered, to be reintroduced to the miscella immediately before spray drying to enhance the flavour and aroma of an instant coffee product for example. Aromatic fractions of interest may be recovered from at least one vessel, or a separate vessel may be provided wherein
25. vegetable material is pre-treated for recovery of aromatic fractions prior to contact and residence to effect extraction in a second vessel. This pre-treatment may be with steam for

example, the steam being passed through the vegetable material feedstock, and condensed for recovery of aromatic fractions. We have also found that a second vessel may be employed to advantage for pre-mixing of suitable solvent and vegetable material feedstock prior to introduction to a main vessel where the major portion of the diffusional mechanisms described hereinbefore take place in a slurry which is then formed into a mat cake or bed on a suitable screen as described before herein, where the major phase separation step is conducted.

In the present invention the diffusional mechanisms to produce a solute containing useful compounds of interest is advanced prior to formation of a vegetable material bed, and the bed itself is of lesser thickness than the typical prior art bed which is typically 1 metre or more in thickness. The apparatus of this invention consists in at least one vessel and an associated screen providing a continuous screen loop with collection chambers associated with the screen and defining phase separation stations thereon. Auxilliary equipment such as screen washing pumping mixing and heating apparatus for example are also provided as required to suit the apparatus and vegetable material feedstock to be processed. Where the solvent is water for example this is typically heated.

Where more polar solvents are used, such as alcohol or hexane for example, the suitable screen and associated collection chambers may be contained in a suitable housing to prevent evaporative loss of such solvents, such housings not being ordinarily necessary when the solvent is aqueous.

Suitable solvents used to extract vegetable material feedstock according to this invention are selected according to the nature of the vegetable material feedstock and the useful compounds to be extracted therefrom, that is, the solubility of 5. the useful compounds or solute, or their miscibility with the solvent, the rule that "like dissolves like" providing a useful guidance. For example vegetable oils consisting mainly of triglycerides of fatty acids may be extracted with hexane, whereas more polar free fatty acids are best extracted with alcoholic 10. solvents. Water is commonly used as a solvent, and may for example be used hot for the extraction of coffee, tea and some barks, while we have found that in other cases it is advantageous to use an aqueous solvent consisting of water to which selected compounds have been added to increase the solubility or misc- 15. ibility of the solute and thus increase the yield and/or quality of the useful compounds extracted.

An apparatus according to this invention is diagrammatically illustrated in a basic form in Figure 4. Referring to Figure 4 the apparatus consists of at least one vessel 1 and a suitable 20. screen 2. The suitable screen is preferably of a type providing a continuous screen loop such as a moving screen filter of the table or disc type, or the drum, or the belt type. The type depicted in Figure 4 is of the belt type which is a preferred type. The suitable screen of a continuous loop type is fitted 25. with collection chambers such as for example collection chambers 3 and 4 preferably provided with a sealing means between chamber and filter as shown for example as 5, 6, 7 in Figure 4, so that the suitable screen may be moved either continuously or intermittently as indicated by arrows 8 and 9.

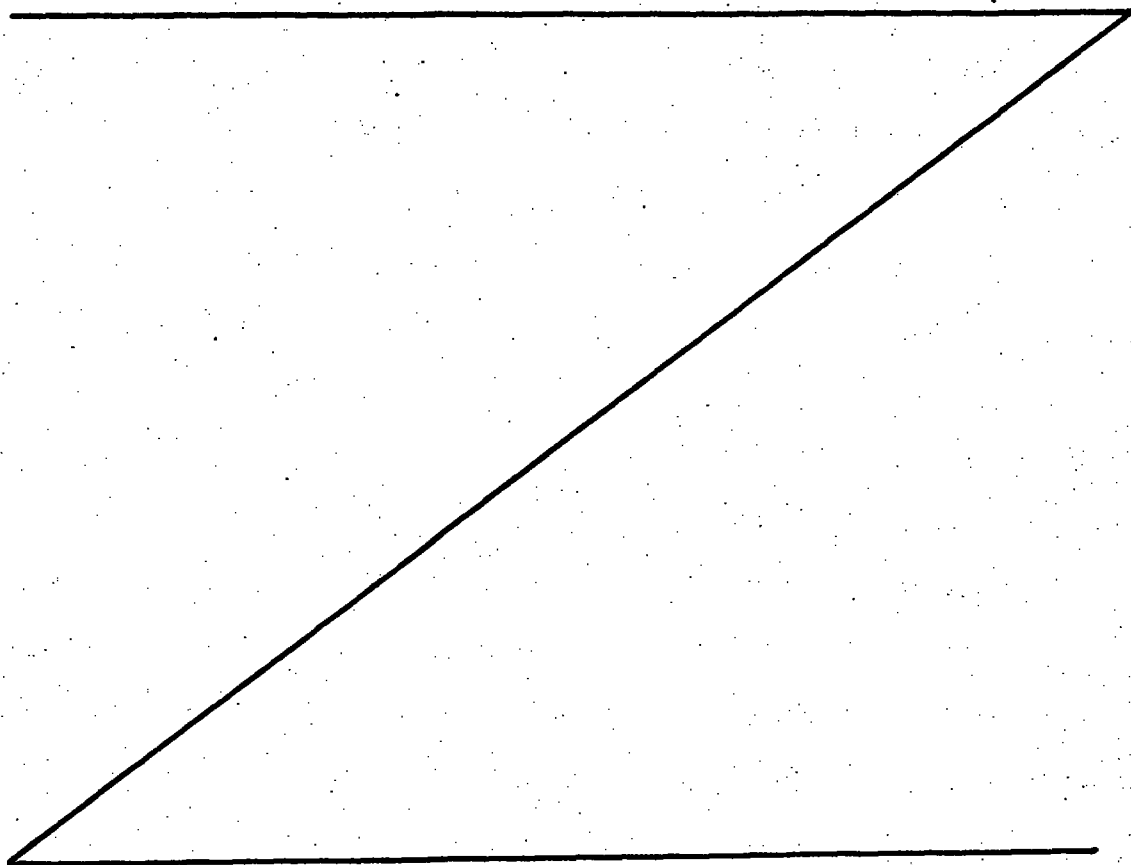
Suitable transfer means 16 are provided between the vessel and the screen to permit the transfer of mixture from the vessel to the screen and the formation of the mat or cake on the screen, after the mixture 5. has been in the vessel for a predetermined or desired period of time.

The transfer means or mechanism can be of any suitable or known type. For example, the transfer means can be a transfer head box as used in the formation 10. of papers and wood fibre insulating boards from an aqueous slurry.

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The principal essence of this form of the invention is that contact between a suitable solvent and the vegetable material feedstock takes place in part in at least one vessel to provide residence time during which extraction proceeds, the suitable solvent and the vegetable material feedstock forming a slurry in the at least one vessel, following which the slurry or mixture is formed in to a mat or cake or bed on the continuous screen, the mat or cake or bed being a bed of the vegetable material through which the suitable solvent is caused to percolate, and characteristically the mat or cake, being the bed is relatively thin when compared with the bed of vegetable material commonly found in most prior art extraction apparatus wherein typically 1 metre or more in depth is used. The bed employed in this form of the invention is relatively thin and relatively large in area when compared with the commonly found prior art bed, and is further characterised in that contact between suitable solvent and vegetable material feedstock takes place prior to formation of the mat or cake, the mat or cake being the bed. In operation comminuted vegetable material preferably comminuted to a particle size which would have a percolation rate of less than 50 litres per hour per bed area 1 square decimetre, per bed 1 metre deep, to take advantage of increased yield and/or increased rate of extraction resulting from the use of decreased particle size (the percolation rate of 50 litres per hour being for a bed without pressure differential) enters at least one vessel 1 where it is mixed with solvent 11 which in the example shown in Figure 4 may contain some solute derived from a washing or solvent recovery station or stage, such as solute containing solvent recovered from chamber 4 in Figure 4.

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The at least one vessel may be a simple open top vessel

1 as shown in Figure 4 or may be a pressure vessel or digester of either the batch or continuous type. Pressure vessels or digesters are known in the lignocellulosic pulp making art 5. which provide the principle of continuous operation, being fitted with feeders and outlet valves enabling maintenance of pressure while at the same time facilitating introduction of vegetable material for treatment and removal of product after treatment. More than one vessel may be used, in which case 10 all vessels may not be alike, each being selected according to function. At the base of vessel 1 slurry consisting of suitable solvent and vegetable material is deposited upon suitable screen 2 so that by virtue of motion of suitable screen as indicated by arrows 8 and 9 a mat cake or bed is formed, the 15. mat cake or bed being transported due to motion of the suitable screen, said motion being intermittent or continuous. Preferably, the motion is continuous. The mat, cake or bed is transported over collection chambers such as collection chambers 3 and 4 and is finally discharged as indicated at 15 20. being practically exhausted of the useful compounds of interest. The mat, cake or bed employed in this invention is preferably less than 1 metre deep, and typically within the range of 8mm to 30mm thick but this is not limiting as it is dependent upon the nature of the vegetable material and the linear speed 25. of the screen for example. Contact between suitable solvent and vegetable material, being referred to as residence time in the art, is accomplished predominantly in the at least one vessel in this invention with the total residence time being contact between suitable solvent and vegetable material

- both in the at least one vessel and on the suitable screen. From experience we have found that the required residence time will vary according to the vegetable material feedstock, the suitable solvent, and the extraction conditions
5. used. It will be appreciated that the residence time needs to be controlled in order to optimise productivity and extract quality and for this reason it is preferred that solvent/vegetable material slurry in the at least one vessel moves in generally plug flow within the vessel so that solvent/
 10. vegetable material slurry formed or charged into that vessel is not mixed with solvent/vegetable material slurry which has remained in that vessel for a time. Thus, with generally plug flow the residence time in the at least one vessel can be determined, being the time taken for solvent/vegetable material
 15. slurry to pass from entry to that vessel, to exit and formation of the mat cake or bed on suitable screen. Paddles may be employed to advantage in the at least one vessel to maintain intimate contact between suitable solvent and vegetable material feedstock especially where the solvent/vegetable
 20. material slurry is heated but such paddles need to be arranged so that the plug flow and hence the residence time are preserved as far as practicable. In this invention, the counter current system as used in most liquid-solid extraction may be preserved by the arrangement of adequate piping.
 25. Still referring to Figure 4, fresh solvent may be sprayed onto the mat, cake or bed 13 by an arrangement of suitable piping 12 so that solvent so sprayed is caused to percolate

through the relatively thin bed (when compared with typical prior art extractors) to be collected in chamber to be conveyed by suitable piping, and a pump where required, to an earlier stage of the apparatus, and in the counter

5. current direction to that of the vegetable material solids. In Figure 4, the solvent sprayed from suitable piping 12 onto bed 13 passes through bed 13 washing solute with it into chamber where the solvent and solute are collected and conveyed in counter current direction by suitable piping 11 to discharge

10. into at least one vessel 1 where it contacts vegetable material also entering at least one vessel at 10 to form new slurry which resides in at least one vessel to complete the cycle. In Figure 4, extract, also termed the miscella in the art being useful compounds contained in solvent is collected in

15. collection chamber 3 being separated from the mat cake or bed on the suitable screen to be discharged 14 for concentration or treatment. As an aid to separation of liquid and solid phases from the bed formed on the suitable screen a pressure differential may be provided across the bed by drawing a

20. partial vacuum on chambers as required. Discharged solids 15 may be pressed if desired to recover underflow therefrom, the underflow being bound or trapped solvent largely depleted of solute by a previous washing stage. Recovered underflow may be returned to the extraction system either by inclusion

25. in the wash or by introduction to the at least one vessel during formation of the slurry for example.

Figure 4 depicts apparatus of this invention in basic form. A more detailed form is shown in Figure 5. Whereas the apparatus depicted in Figure 4 has two phase separation

stations as defined by the two collection chambers, it is preferred that several phase separation stations be provided, and by way of example only five phase separation stations are shown in Figure 5. Extraction apparatus functions which may be simply and effectively performed using the versatile apparatus of this invention, by provision of adequate piping and auxiliary equipment will now be described with reference to Figure 5. In Figure 5 two vessels are illustrated, associated with a suitable screen of the continuous belt type. The two vessels 25 and 26 are used to prepare a slurry consisting of suitable solvent and vegetable material feedstock, and to provide residence time during which the diffusional mechanisms described hereinbefore take place. Vessel 25 typically smaller than vessel 26 is fitted with mixer 27 and is used to thoroughly mix vegetable material 28 and weak solute 29 to produce a slurry which is discharged into vessel 26 and shown at 30. Vessel 26 contains the slurry produced in vessel 25 for a substantially pre-selected residence time related to the vegetable material diffusivity and particle size as described hereinbefore, and during residence in vessel 26, the slurry moves in generally plug flow as shown by arrow 31, so that when the pre-selected residence time has elapsed the slurry discharges onto screen 32 as shown to form a mat cake or bed 33 which is displaced by movement of screen 32 as indicated at 34 to pass in sequence over collection chambers 35, 36, 37, 38, 39, defining phase separation stations, until discharged at 40 being largely exhausted of useful compounds.

Suitable solvent enters the extraction system to be sprayed onto mat cake or bed as shown at 41 and percolates through mat cake or bed to be collected in collection chamber 39, and accumulated in receiver 42 prior to delivery by pump 43 to be sprayed onto mat cake or bed as shown at 44. Solute containing solvent sprayed onto the mat cake or bed at 44 percolates through this to be collected in collection chamber 38 and accumulated in receiver 46 prior to delivery by pump 47 to be sprayed onto mat cake or bed as shown at 48. The sprayed solute again percolates through the mat cake or bed to be collected in collection chamber 37 and accumulated in receiver 49 prior to delivery by pump 50 to mixing vessel 25 as shown. Thus the solvent passes through the mat cake or bed three times in this example to be delivered to vessel 25 in accordance with the general principal of counter current washing and extraction. To further illustrate the versatility of the apparatus of this invention, an example wherein one of the phase separation stations, defined by collection chamber 35 is arranged to remove fines to minimise suspended solid in the final miscella or extract which is collected in collection chamber 36. Solute or liquid phase which passes through the screen at the phase separation station defined by the collection chamber 35 at the time the mat cake or bed is formed is re-cycled to remove suspended solids therefrom the solute or liquid phase is collected in collection chamber 35 and accumulated in receiver 51 to be pumped by pump 52 and either sprayed onto the mat cake or bed at 53 or passed back into the main slurry stream 54 as shown at 55.

Re-cycling solute or liquid phase as shown uses the

mat cake or bed itself as a fines filter by causing suspended solids to be trapped therein. In Figure 5, the phase separation station defined by collection chamber 35 is used to remove suspended solids from the miscella while

5. concurrently, displacement of the screen 32 as indicated by arrow 34 causes the miscella to be transported by the mat cake or bed, to be again separated at the phase separation station defined by collection chamber 36 leaving the suspended solids substantially trapped in the mat cake or bed. The miscella,

10. being suitable solvent containing solute, that is useful compounds extracted from the vegetable material feedstock is accumulated in receiver 57 to be delivered by pump 58 as the weak extract which may be concentrated or otherwise treated as appropriate for the particular extract and intended

15. end use thereof. The weak extract, is also known in the art as miscella and also as overflow. The vegetable material feedstock, after being largely exhausted of useful compounds is discharged as shown in Figure 5. This discharged and largely exhausted vegetable material, sometimes referred to as Regectamenta in the

20. art also contains some bound or trapped solvent, largely depleted of solute by a previous washing stage, and sometimes referred to in the art as underflow. This underflow may if desired be also recovered and returned to the extraction system, and by way of example, the recovery of underflow is illustr-

25. ated in Figure 5. Regectamenta, being largely exhausted vegetable material is pressed in a suitable press 60 such as a batch hydraulic press or a continuous screw press wherein further phase separation is conducted to remove or recover underflow⁶¹ therefrom which may be returned to a preceding treatment stage such as the wash stage or the slurry preparation

stage which takes place in vessel 25. The Rejectamenta after removal of underflow, may be discharged as at 65 to be used for example as a boiler fuel or plant growing medium base for example, depending upon the nature of the original vegetable material feedstock, and economic factors.

It will be appreciated that this invention provides method and a-paratus for the extraction of useful compounds from vegetable material feedstock which utilises advantages to be gained by comminuting the vegetable material feedstock to relatively small particles when compared to particle sizes commonly used in the prior art. The broad invention encompasses the extraction of useful compounds from vegetable material generally and within this scope the conditions of extraction may be selected to suit the particular vegetable material feedstock and extract product to be manufactured.

By way of example, the employment of this invention for the manufacture of a coffee extract as used to produce so called instant coffee will now be described.

Coffee beans are roasted and ground as in the prior art, with the exception that the particle size is reduced to less than 2mm (diameter according to Rosin, Rammler and Sperling) whereas the typical particle size used in the prior art may be near 3mm to obtain an adequate percolation rate. Preferably, the particle size is less than 1mm (Rosin, Rammler and Sperling) so that full advantage can be taken of the increased yield and/or increased extraction rate as disclosed hereinbefore. The particle size of less than 1mm will be found to produce a percolation rate which is too low for use in prior art Rotocell extractors for example.

It is preferable but not essential to first recover the aromatic fraction from a coffee extract in order that this is not lost during the main extraction sequence, the aromatic fraction being returned to the main stream again immediately prior to concentration such as spray drying for example. The aromatic fraction may be recovered by treatment of the ground coffee with live steam for example, the live steam being collected and condensed so that the aromatic fraction is contained in the condensate. The ground coffee may be treated with live steam either before or after preparation of the slurry required in the process of this invention. For example the treatment may take place in a vessel wherein live steam is introduced to the dry ground coffee particles or a zone may be provided in a vessel where live steam is injected into a slurry in a main vessel during the residence time therein. The suitable solvent used in the case of coffee extraction is heated water, preferably heated to at least 90°C and more preferably to within the range 90°C and 100°C . Higher temperatures may be used by accommodating the suitable screen and associated collection chambers in a suitable closure and by sealing a main vessel in the form of a digester as used for example in the art of lignocellulosic pulp manufacture.

Input to a main vessel, in the form of a coffee grounds in water or weak extract slurry or dry coffee grounds and water or weak extract is adjusted in relation to the slurry output from such vessel so that the slurry, in generally plug flow through the vessel, completes a pre-determined residence time as indicated by temperature conditions and fineness of grind of the coffee. A mat, cake or bed is formed from the

slurry, and on a suitable screen and as generally described in the examples associated with Figure 4 and Figure 5 herein and following phase separation steps by successively passing the mat, cake or bed over phase separation stations, generally as

5. described in Figure 4 and Figure 5 herein a coffee miscella is recovered. The bed, being the mat or cake formed on the suitable screen from the slurry is characteristically thinner than the typical prior art bed, being less than 1 metre in depth and more typically between 4mm and 50mm in depth.
10. The recovered coffee miscella may be concentrated as known in the art by for example evaporation in a partial vacuum or reverse osmosis process, and may be dried as required by for example spray drying or freeze drying to produce instant coffee powder. Where an aromatic fraction has been recovered prior
15. to execution of the main extraction this aromatic fraction is re-introduced to the main stream miscella prior to the final drying step to produce an instant coffee powder with superior aroma and flavour. It can be seen from reference for example to Figure 5 that hydrolysis steps may be introduced if
20. desired and as sometimes practiced in liquid-solid extraction, wherein the solid is a vegetable material and it is desired to render compounds contained in the vegetable material more soluble. Such hydrolysis steps may be included at any of the phase separation stations defined by the screen collection
25. chambers for example, or may take place in a vessel prior to formation of the mat, cake or bed. Hydrolysis steps are sometimes used in coffee and tea extraction to increase the solubility of certain carbohydrates contained in these vegetable materials.

By way of further example, the employment of this invention for the manufacture of a bark extract as used to produce a substantially polyphenolic substance will now be described. Substantially polyphenolic bark extracts are used as tanning agents and in the manufacture of some bonding and sizing agents for example.

The extraction conditions used to prepare bark extracts varies, depending upon the tree species from which the bark is derived. For example, bark from the Wattle species *Acacia mearnsii* can be simply extracted using hot water as a solvent while bark from the Pine species *Pinus Radiata* is more difficult to extract. *Acacia mearnsii* bark produces high yields of extract using hot water as a solvent while the yields of extract from *Pinus radiata* bark using hot water as a solvent are low, with unfavourable economics as a consequence. In this example the extraction of *Pinus radiata* bark according to this invention will be described.

The extraction of polyphenolic material from the bark of *Pinus radiata* is preferably performed while the bark is in a green condition, that is to say, the bark is removed from the log while sap is still present, the bark being fresh and still containing some sap or natural moisture at the time the extraction process is performed. A reason for this is illustrated in Figure 6 herein.

To prepare Figure 6 *Pinus radiata* bark was extracted at various intervals after removal from the freshly felled log and the extract yield was determined. It can be seen from Figure 6 that at least with *Pinus radiata* bark, it is an advant-

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age to perform the extraction within a reasonable time after removal from the fresh log.

We have found that the *Pinus radiata* bark should be extracted while in a green or substantially green condition, and
5. preferably at least within 100 days of removal from the fresh log. Figure 6 shows that it is more preferable to process the *Pinus radiata* bark as soon as practicable after removal from the fresh log, with a significant advantage being shown when the bark is extracted within 30 to 40 days and an
10. even greater advantage when the bark is extracted within 15 to 20 days.

The preferred extraction conditions for preparing substantially polyphenolic extracts from the bark of *Pinus radiata* are disclosed in New Zealand patent specification No. 179933,
15. and these preferred extraction conditions can be simply and effectively provided using the method and apparatus of this invention. An advantage of this invention however is that the bark may be more finely comminuted than is desirable for processing in prior art apparatus such as the Rotocell or De Smet extractor for
20. example, the advantage being increased yield and/or increased rate of extraction, due to the employment of the improved diffusional phenomena disclosed hereinbefore without the limitations imposed by reduced percolation rates.

The process of extracting useful compounds from bark,
25. such as *Pinus radiata* bark requires that the bark be comminuted to small particles. This comminution may be a single step, or conveniently may be a multiple step operation. For convenience in handling we have found it to be advantageous to employ a first step wherein the bark is hammermilled

but this is not essential. It will be appreciated however that where large quantities are to be processed it is an advantage to have a consistent product which can be reliably transported by conveyor and the like in the extraction

5. operation.

To reduce the bark to a particle size suitable for extraction using the method and apparatus of this invention, a disc refiner has been found suitable, although other forms of attrition apparatus may also be used. Suitable disc

10. refiners are for example the Bauer or Sprout Waldron as commonly used in the wood pulp industry and the bark is preferably comminuted in the presence of water which may remain with the bark, at least in part to become the suitable solvent in the extraction process.

15. The bark may be comminuted for example to a fineness which is characterised by a percolation test.

The percolation test is that for a bed of the comminuted bark 1 metre deep the percolation rate of water heated to 90 °C through the bed is less than 50 litres per hour per square

20. decimetre of bed area.

We have found that the bark may be comminuted to a fineness having a percolation rate of less than 10 litres per hour per square decimetre of bed area.

We have for example refined *Pinus radiata* bark in a double
25. disc refiner, the resultant product having a percolation rate of only 0.8 litres per hour when tested as described and being entirely unsuitable for use in the prior art Carrousel extraction apparatus.

In extracting the compounds from the vegetable matter, and as stated hereinbefore, it is preferable that the particles of vegetable matter be commutated, or disintegrated into a fine or particulate state, the particles preferably being up to 4mm in size.

During extraction, it is preferred that the aqueous solution or said at least one solvent, which is usually water, include one or more stabilising compounds, such as for example sodium sulphite, sodium bisulphite, sodium metabisulphite sulphur dioxide, sulphuric acid, potassium sulphite, potassium bisulphite, potassium metabisulphite and ammonium sulphite.

In addition, it is preferred that the aqueous solution also include one or more extraction compounds which can assist in the extraction of the useful compounds from the vegetable matter, such extraction compounds being for example, sodium carbonate, sodium bicarbonate, potassium carbonate, sodium hydroxide, potassium hydroxide, ammonium hydroxide, ammonium carbonate, ammonia, sodium sulphite, sodium bisulphite, and sodium metabisulphite.

In preferred forms of the invention, the aqueous solution or solvent will include one or more stabilising compounds as well as one or more extraction compounds.

It is preferred that during extraction and by way of example only, the temperature of the aqueous solution or solvent, at least within the extraction vessel, and if desired during application while the mat is on the screen, is elevated to above ambient, but to be less than 100°C.

In one form of the invention, the temperature is between 40°C and 90°C being preferably between 60°C and 90°C.

- A preferred residence time, that is to say the time during which the vegetable matter is subjected to the
5. aqueous solution (in this form of the invention - both within the vessel and on the screen], is up to approximately 120 minutes.

In one form of the invention however, the extraction time is preferably less than 60 minutes.

10. The preferred form of the invention results in the compound or phenolic extract resulting from the extraction being stored or used, and in one form of the invention, it is preferably concentrated prior to storage or use, such as by evaporation. Alternatively, a phenolic extract can be concentrated to dry solids.

15. These are however by way of example.

One preferred method of extraction is generally as described and claimed in our New Zealand patent specification No. 179933.

- It will be appreciated that the method and apparatus
20. of this invention provides for the extraction of useful compounds from a wide variety of materials of vegetable origin, such as the examples leaf, bark, root, stem, seed and Blossom, and that these useful compounds have many applications in the manufacture of beverages, pharmaceutical products, adhesives,
25. tanning agents, spices, dyes and fragrances for example. Accordingly, and depending upon the nature of the useful compounds and the intended end use thereof, they may be suitably treated or modified, for example, for the purpose of improving

or enhancing properties, or to facilitate use, transportation or storage. Such suitable treatment may for example include hydrolysis, either acidic or basic, or a concentration step involving osmosis, ultra-filtration or evaporation, or other appropriate treatment.

The particular examples given herein of the use of the method and apparatus of this invention, for the extraction of useful compounds from varieties of vegetable material, namely the recovery of tea, coffee, and bark extracts, have in common that these extracts are usually concentrated to dryness, the dry extract product, in powder or granular form being for example the instant tea and coffee of commerce, and a polyphenolic powder substance which may be used in adhesive and tanning applications. Methods practiced in the art to concentrate these extracts to dryness include freeze drying and spray drying processes for example, and extracts prepared according to this invention may be so treated when appropriate, or treated in some other manner as desired.

It will be appreciated that modifications and improvements may be made to this invention without departing from the scope thereof, as defined by the appended claims.



CLAIMS

1. A method of extracting useful compounds from vegetable matter, (as herein defined), said method including the steps of:

- 5 (1) preparing a mixture of the vegetable matter and at least one solvent;
- (2) allowing said mixture to reside in at least one vessel for a substantially predetermined time;
- 10 (3) thereafter transferring the mixture from said at least one vessel to a screen so that a mat or cake is formed on said screen, whereby the solvent phase separates from the vegetable phase by passing through said screen;
- 15 (4) recovering the solvent phase and obtaining useful compounds extracted from the vegetable matter as a filtrate from said screen.

2. A method as claimed in claim 1, wherein at least one solvent is applied to said mat or cake on said screen.

20 3. A method as claimed in either of the preceding claims, wherein said solvent phase containing useful compounds extracted from said vegetable material, passes into at least one collection chamber provided adjacent said screen.

4. A method as claimed in claim 3, wherein a plurality
25 of collection chambers are provided adjacent said screen.

5. A method as claimed in either of the preceding claims 3 or 4, wherein said screen is movable, relative to said chamber(s).

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6. A method as claimed in any one of the preceding claims 1 to 5, wherein the mat or cake is less than 1 metre in depth.
7. A method as claimed in any one of the preceding claims 1 to 6, wherein the mat or cake is between 8mm and 30mm in depth.
8. A method as claimed in any one of the preceding claims wherein the particle size of the vegetable matter is such as to enable solvent to pass through said mat or cake at a rate of less than 10 litres per square decimeter per hour.
9. A method as claimed in any one of the preceding claims, wherein the vegetable matter is in particulate form having a particle size of up to 4mm.
10. A method as claimed in any one of the preceding claims, wherein said at least one solvent is heated.
11. A method as claimed in any one of the preceding claims, wherein said at least one solvent is heated to a temperature of less than 100°C.
12. A method as claimed in either of the preceding claims 10 and 11, wherein the temperature of said at least one solvent is between 40°C and 90°C.
13. A method as claimed in any one of the preceding claims, wherein the residence time (as herein defined) for contacting said vegetable matter and said at least one solvent is up to 120 minutes.
14. A method as claimed in claim 13, wherein the residence time is less than 60 minutes.
15. A method as claimed in any one of the preceding claims, wherein the vegetable matter is coffee beans.



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16. A method as claimed in claim 15, wherein the coffee beans are in particulate form, having a particle size of less than 2mm.
17. A method as claimed in claim 16, wherein the particle
5 size is less than 1mm.
18. A method as claimed in any one of the preceding claims 15 to 17, wherein the coffee beans are processed to recover aromatic fraction.
19. A method as claimed in claim 18, wherein the aromatic
10 fraction is recovered by treatment of the coffee beans with live steam, said steam being collected and condensed such that the aromatic fraction is contained in condensate form.
20. A method as claimed in either of claims 18 and 19, wherein the coffee beans are treated with live steam before
15 or during, contact with said solvent.
21. A method as claimed in any one of the preceding claims 1 to 14, wherein the vegetable matter is bark.
22. A method as claimed in claim 21, wherein the bark is in a green or substantially green condition (as defined herein).
- 20 23. A method as claimed in either of claims 21 and 22, wherein the solvent includes at least one stabilising compound.
24. A method as claimed in any one of the preceding claims 21 to 23, wherein the solvent includes at least one extraction compound.
- 25 25. A method as claimed in any one of the preceding claims 21 through 24, wherein the solvent includes at least one stabilising compound and at least one extraction compound.
26. A method as claimed in claim 1, substantially as hereinbefore described.