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(54) Title: PROCESSES FOR FRACTIONATING PHOSPHOLIPIDS

(57) Abstract: Processes of fractionating phospholipids are disclosed. In one embodiment, the process includes placing a phospholipid containing material in contact with an adsorbent, such that the adsorbent associates with at least one phospholipid of the phospholipid containing material. The process may further include eluting the at least one phospholipid from the adsorbent.

PROCESSES FOR FRACTIONATING PHOSPHOLIPIDS

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TECHNICAL FIELD

[0001] The present invention relates generally to phospholipids and more particularly, to processes of purifying and/or fractionating phospholipids.

BACKGROUND OF THE INVENTION

[0002] Lecithin is a commercial by-product of vegetable oil production and is typically obtained from vegetable oils such as soybean oil, canola oil, sunflower oil, or corn oil. The high phospholipid content of lecithin makes it a natural emulsifier, stabilizer, baking improver, and wetting agent.

[0003] The mains phospholipids in lecithin are phosphatidyl choline (PC), phosphatidyl ethanolamine (PE), phosphatidyl inositol (PI), and phosphatidic acid (PA). The purified phospholipids, in particular PC, are often desired since they are value added nutritional and/or pharmaceutical compounds. Various methods have been developed to purify these phospholipids.

[0004] Some of the current methods of purifying the individual phospholipids use lecithin as a starting point. While these methods may be effective at purifying the phospholipids from the lecithin, the resulting products are the purified phospholipids and "phospholipid depleted" lecithin. A drawback of these methods is that while a higher value phospholipid may be obtained, the value of the lecithin itself may be of lower value since it has been depleted of the high-value phospholipid.

[0005] Thus, needs exists for improved methods of fractionating and/or purifying phospholipids, yet keeping the value of the various products associated with the process.

SUMMARY OF THE INVENTION

[0006] In each of its various embodiments, the present invention fulfills these needs and discloses processes of fractionating phospholipids.

[0007] In one embodiment, a method of fractionating phospholipids comprises placing a phospholipid containing material in contact with an adsorbent, such that the adsorbent associates with at least one phospholipid of the phospholipid containing material.

[0008] In each of the various embodiments of the present invention, the adsorbent may be a polymeric resin and may be selected from the group consisting of a phenolic resin, a styrene resin, a non-ionic resin, an acrylic resin, a cation-exchange resin, a weak-base resin, and combinations of any thereof. In each of the embodiments, the polymeric resin may also be a styrene resin or a phenolic resin.

[0009] In each of the embodiments of the present invention, the phospholipid containing material may be selected from the group consisting of miscella, molasses, lecithin, and combinations of any thereof. In the various embodiments, the phospholipid containing material may be miscella.

[0010] The method of fractionating phospholipids of the present invention may also include eluting the at least one phospholipid from the adsorbent which may be done by comprises an elution solvent with the adsorbent. The phospholipid containing material may contain less than 5% by weight of phospholipids and the at least one phospholipid eluted from the adsorbent contains more than 50% by weight of the phospholipids.

[0011] The method of fractionating phospholipids of the present invention may also include placing the at least one phospholipid eluted from the adsorbent in contact with a second adsorbent, which may optionally include mixing the at least one phospholipid eluted from the adsorbent with a solvent before placing in contact with the second absorbent. The methods of fractionating phospholipids of the present invention may further comprise collecting the eluted, at least one phospholipid.

[0012] The methods of fractionating phospholipids of the present invention may further include mixing a solvent with the phospholipid containing material, thus producing a feed, where placing the phospholipid containing material in contact with the adsorbent comprises placing the feed in contact with the adsorbent. The methods may further comprise eluting the at least one phospholipid from the adsorbent with an elution solvent and/or collecting the eluted, at least one phospholipid and the elution solvent. The methods may also allow a precipitate to form in the eluted, at least one phospholipid and the elution solvent, and may include removing the precipitate from the eluted, at least one phospholipid and the elution solvent and concentrating the at least one phospholipid in the elution solvent.

[0013] In each of the various embodiments of the present invention, the solvent may selected from the group consisting of an alcohol, an alkane, water, and combinations of any thereof and the at least one phospholipid may be selected from the group consisting of phosphatidyl choline (PC), phosphatidyl ethanolamine (PE), phosphatidyl inositol (PI), phosphatidic acid (PA), and combinations of any thereof.

[0014] The methods of the present invention may also include extracting the phospholipid containing material from a phospholipid source, adjusting a pH of the phospholipid containing material to a pH of between 8.5-10.0, and/or removing solids from the phospholipid containing material.

[0015] In the various embodiments of the present invention, the adsorbent may located in a simulated moving bed chromatography system.

[0016] In various embodiment, the solvent used in the present invention may be the alkane and selected from the group consisting of hexane, heptane, or a combination thereof.

[0017] The phospholipids of the present invention may originate from a source selected from the group consisting of soybean, egg, canola, sunflower, corn, and combinations of any thereof.

[0018] In the present invention, a process of fractionating phospholipids includes mixing a phospholipid containing material selected from the group consisting of miscella, lecithin, molasses, and combinations of any thereof with a solvent, thus producing a feed, placing the feed in contact in contact with a polymeric resin, such that the polymeric resin associates with at least one phospholipid of the phospholipid containing material, and eluting the at least one phospholipid from the polymeric resin with an elution solvent.

[0019] The polymeric resin used in the present invention may be selected from the group consisting of a phenolic resin and a styrene resin, and the solvent may comprise an alcohol, an alkane, water, and combinations of any thereof. The elution solvent may comprises an alcohol, an alkane, water, and combinations of any thereof.

DESCRIPTION OF THE DRAWINGS

[0020] Figure 1 is a flowchart of one embodiment of a process for fractionating phospholipids of the present invention.

[0021] Figure 2 is a flowchart of another embodiment of a process for fractionating phospholipids of the present invention.

[0022] Figure 3 shows a graph illustrating the ability of an embodiment of a process of the present invention to fractionate phospholipids.

[0023] Figure 4 shows another graph illustrating the ability of another embodiment of a process of the present invention to fractionate phospholipids.

[0024] Figure 5 shows an additional graph showing the ability of a further embodiment of a process of the present invention to fractionate phospholipids.

[0025] Figure 6 depicts a graph showing the ability of yet another embodiment of a process of the present invention to fractionate phospholipids.

[0026] Figure 7 is a graph showing the ability of one embodiment of the present invention to fractionate phospholipids and separate the phospholipids from sterols.

[0027] Figure 8 shows a graph illustrating the ability of an embodiment of a process of the present invention to fractionate phospholipids.

[0028] Figure 9 depicts a graph showing the ability of yet another embodiment of a process of the present invention to fractionate phospholipids.

[0029] Figure 10 shows another graph illustrating the ability of another embodiment of a process of the present invention to fractionate phospholipids.

[0030] Figure 11 is a graph showing the ability of one embodiment of the present invention to fractionate phospholipids.

[0031] Figure 12 illustrates one embodiment of a simulated moving bed chromatography system set-up to perform a process of fractionating phospholipids of the present invention.

[0032] Figure 13 is a graph showing the ability of another embodiment of the present invention to fractionate phospholipids. The x axis is Bed Volumes and the y axis is concentration of the phospholipids in mg/Kg.

[0033] Figure 14 illustrates another embodiment of a simulated moving bed chromatography system set-up to perform a process of fractionating phospholipids of the present invention.

[0034] Figure 15 depicts a graph showing the ability of one embodiment of the present invention to fractionate phospholipids.

[0035] Figure 16 shows an additional embodiment of a simulated moving bed chromatography system set-up to perform a process of fractionating phospholipids of the present invention.

[0036] Figure 17 is a flowchart of a two-stage process of an embodiment of fractionating phospholipids of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0037] Processes of fractionating and/or purifying phospholipids are disclosed. In one embodiment, the process may include placing a phospholipid containing material in contact with an adsorbent, such that the adsorbent associates with at least one phospholipid of the phospholipid containing material. The process may further include eluting the at least one phospholipid from the adsorbent. The adsorbent may be a resin.

[0038] The process may further include mixing a solvent with the phospholipid containing material, thus producing a feed and placing the feed in contact with the adsorbent. The process may also include collected the eluted, at least one phospholipid. The adsorbent or resin may also be placed in a column.

[0039] The phospholipid containing material may be miscella, molasses, lecithin, or combinations of any thereof and may originate from a source such as soybean, egg, canola, sunflower, corn, or combinations of any thereof.

[0040] In a further embodiment, the process may include extracting the phospholipid containing material from a phospholipid source. The process may further comprise removing solids from the phospholipid containing material.

[0041] In yet a further embodiment, the adsorbent may be located in a simulated moving bed chromatography system.

[0042] In an additional embodiment, the phospholipid containing material may contain less than 5% by weight of phospholipids and the at least one phospholipid eluted from the adsorbent may contain more than 50% by weight of the phospholipids.

[0043] The phospholipid containing material may be any substance or compound capable of having phospholipids extracted or fractionated therefrom using a resin. To be placed in contact with the resin, the phospholipid containing material may be in a liquid form and/or suspended in a liquid. The phospholipid containing material may also originate from and/or be extracted from a phospholipid source. For example, a phospholipid containing material such as lecithin may be extracted from a phospholipid source such as soybeans. The lecithin may be crude lecithin, de-oiled lecithin, lecithin paste, or any other lecithin source. Examples of phospholipid sources include without limitation eggs or fractions thereof, and vegetable crops such as soybean, canola, sunflower, or corn or fractions thereof.

[0044] In an additional embodiment, the phospholipid containing material is not lecithin, and may be miscella, solubles, or molasses.

[0045] In one embodiment, the phospholipid containing material originates from vegetable, i.e., soybean, processing. Figure 1 illustrates a flowchart of one embodiment of a process used to obtain a phospholipid containing material and fractionate the phospholipids therefrom. Any of the various products of Figure 1 that contain phospholipids and are capable of having the phospholipids fractionated or purified from the product using an adsorbent may be used in the processes of the present invention.

[0046] Figure 1 also depicts one embodiment of the present invention where the phospholipid containing material comprises soy miscella, but in another embodiment, the phospholipid containing material may comprise crude lecithin. In Figure 1, the soy miscella is mixed with the solvent, hexane or ethanol, and placed in contact with a resin to fractionate the phospholipids.

[0047] In Figure 1, the soy miscella is obtained from the solvent extraction of full-fat soy flakes and may include soy oil, phospholipids, extraction solvent, other impurities extracted from the soybean along with the oil and solvent (i.e., possibly sugars from the soy and/or other solids from the soy flakes), and combinations of any thereof.

[0048] In various embodiments of Figure 1, the different phospholipid containing material (i.e., soy miscella or crude lecithin) may use different resins and/or solvents to fractionate the phospholipids. For instance, in one embodiment, ethanol extracted soy miscella may be applied to a column (or simulated moving bed system) with a non-functional poly-styrene resin, rinsed with ethanol, and eluted with hexane or hexane and ethanol. In another embodiment, crude lecithin extracted with hexane may be placed in contact with a non-functional phenolic based resin, rinsed with hexane, eluted with ethanol or a combination of ethanol with hexane, and rinsed with hexane. This embodiment may yield a fractionated product having at least 45% phosphatidyl choline.

[0049] In the various processes for fractionating phospholipids described herein, a solvent may be mixed with the phospholipid containing material to produce a feed. The feed may be placed in contact with the resin, wherein the resin associates with a phospholipid of the phospholipid containing material. The solvent may be: an alkane including, but not limited to, hexane, heptane, or a combination thereof; an alcohol, water; or combinations of any thereof. The alcohol may be ethanol, isopropyl alcohol, beverage grade ethanol, denatured ethanol, or combinations of any thereof.

[0050] In another embodiment shown in the flowchart of Figure 2, the phospholipid containing material may be soy miscella (option A) or soy molasses (which may also be referred to as soy solubles) (option B). Soy solubles or soy molasses refers to a de-solventized aqueous ethanol extract (or desolventized and evaporated soy miscella) that may be obtained from the production of soy protein concentrate. In a further embodiment, the phospholipid containing material may be soy miscella which is in essence soy solubles or soy molasses that has not been de-solventized. Option A and option B are very similar, where option B adds water and ethanol to the soy molasses prior to pH adjustment.

[0051] In one embodiment, the phospholipid eluted from the adsorbent is collected. The phospholipid that is collected may be further purified, concentrated, or further processed to produce a finished product. The phospholipid may be eluted with an elution solvent. The elution solvent may be selected from the group consisting of: an alkane including, but not limited to, hexane, heptane, or a combination thereof; water; an alcohol; or combinations of any thereof. The alcohol may be ethanol, isopropyl alcohol, beverage grade ethanol, denatured ethanol, or combinations of any thereof.

[0052] In one embodiment, the adsorbent or resin may be placed in a chromatography column where the phospholipid containing material or the feed is placed in contact with the resin. In such manner, a phospholipid, such as phosphatidyl choline (PC), phosphatidyl ethanolamine (PE), phosphatidyl inositol (PI), phosphatidic acid (PA), or combinations of any thereof, may associate with the resin and as a solvent or other liquid is run through the resin, other components of the phospholipid containing material or the feed are washed away from the phospholipid, thus, purifying or fractionating the phospholipid from the phospholipid containing material. In an additional embodiment, the resin may be placed into a simulated moving bed chromatography system.

[0053] The resin of the present invention may be a polymeric resin such as a non-ionic resin, a phenolic resin, a styrene resin, an acrylic resin, a cation-exchange resin, a weak-base resin, or combinations of any thereof.

[0054] In a further embodiment, a phospholipid containing material or a feed including the phospholipid containing material and/or a solvent may be pH adjusted such as to a pH of between 4.5-6.5, a pH of between 8.5-10.0, or a pH of between 9.0-9.5, and placed into contact with the resin. In other embodiments, the phospholipid containing material or a feed including the phospholipid containing material may have solids removed therefrom and placed into contact with the resin.

[0055] The following exemplary, non-limiting examples are provided to further describe the embodiments presented herein. Those having ordinary skill in the art will appreciate that variations of these Examples are possible within the scope of the invention.

[0056] Example 1.

[0057] Adsorption and desorption of phospholipids was done on a Mitsubishi WK10 acrylic weak acid cation-exchange resin. 100 mls of the resin were loaded into a 15 mm diameter glass column. The resin was rinsed with 3 bed volumes (BV) of isopropyl alcohol (IPA), followed by rinsing the resin with 3 BV of hexane.

[0058] A feed was prepared by dissolving de-oiled soy lecithin in hexane at a 1:1 mass ratio. The feed was run over the resin in the 100 ml column. The basic load/elute test of this Example included 10 BV of the hexane/lecithin feed applied to the column, followed by 3 BV of hexane, followed by 3.5 BV of isopropyl alcohol. All runs were performed at 42°C and a rate of 5 mls/minute. The column effluent was fractionated to determine the profile and the results of such fractionation are shown in Table 1. The column produced 1.16 grams of phosphatidyl choline (PC). A composite purity of the PC was calculated to be 82% as determined by dividing the total amount of PC by the sum of the PC, phosphatidyl ethanolamine (PE), and the phosphatidyl inositol (PI) represented by the following equation: PC/(PC + PE + PI).

[0059] Table 1.

Sample ID	PE mg/kg	PC mg/kg	PI mg/kg	Total mg/L	Total %	%PC
Feed	105235	113199	60684	279118	27.9	41
Raffinate & rinse	86050	92192	48032	226274	22.6	41
Product fraction 1	331	1113	25	1469	0.1	76
Product fraction 2	4734	20193	0	24928	2.5	81
Product fraction 3	568	3050	0	3618	0.4	84
Product fraction 4	273	1529	13	1815	0.2	84
Product fraction 5	97	900	26	1024	0.1	88

[0060] The enhancement purity of the various phospholipids is shown in Figure 3 and is represented by the percent of PC relative to the other phospholipids (PLs) in the column product obtained from a similar study.

[0061] Example 2.

[0062] Pulse testing for chromatographic separation of phospholipids was performed with Dow XAD761 non-functional phenolic resin (a phenol-formaldehyde condensate). 100 mls

of the resin was loaded into a 15 mm glass column. The resin was washed with 3 BV of IPA followed by 3 BV of hexane. The feed was prepared by dissolving crude soy lecithin in hexane at a 1:1 mass ratio and mixing thoroughly. 2 mls of the feed was contacted with the 100 mls of the resin. After applying the feed to the resin, 1 BV of hexane was applied to the column and followed by 3.5 BV of an ethanol elution. This Example was performed at ambient temperature and a flow rate of 2.5 mls/minute. The effluent history of this Example is shown in Figure 4.

[0063] Example 3.

[0064] Affinity chromatography evaluation of the Dow FPA54 weak base phenolic resin (anion exchange resin based on phenol-formaldehyde matrix) on soy lecithin was performed. 100 mls of the resin was loaded into a 15 mm glass column and washed with 3 BV of IPA, followed by 3 BV of hexane. The feed was prepared by dissolving crude soy lecithin in hexane at a 2:1 mass ratio and thorough mixing. 2.5 mls of the feed was contacted with the 100 ml column of resin. After the feed was passed through the resin, 1.5 BV of hexane was passed over the column followed by 4 BV of ethanol. The Example was performed at ambient temperature and a flow rate of 2.5 mls/minute. The results indicate some separation of PC from the PE and the effluent history is shown in Figure 5.

[0065] Example 4.

[0066] Lecithin paste was dissolved at a ratio of 50/50 in a solution of 70% hexane/30% ethanol. 10 mls of this feed was contacted with a Dow XAD761 non-functional phenolic resin, followed by a 70% hexane/30% ethanol mobile phase elution. The results of this Example are shown in Figure 6 and show that there is a separation between PC and PE and PI.

[0067] Example 5.

[0068] A solvent comprising 70% hexane/30% ethanol was prepared. This solvent was mixed with soy solubles at a ratio of 50/50 and mixed for 30 minutes. The solution was allowed to phase separate and the light (organic) phase was analyzed for phospholipid content. The results of such analysis are shown in Table 2. The results indicate a greater than 97% phospholipid extraction of the soy solubles.

70/30 hexane/ethanol	Percent extracted			PC/(PC+PE+PI)
	PE	PC	PI	
Extract, %	98.6	97.8	100.0	-
Feed (ppm)	6556	10162	7798	37%
Organic	14137	15087	11843	37%

Aqueous	202	346	0	63%

[0069] Example 6.

[0070] 100 mls of Dow XAD761 was loaded into a 15 mm glass column and washed with 3 BV of 70% hexane/30% ethanol. A feed was prepared by extracting soy solubles (30% dry solids) with a 70% hexane/30% ethanol solution at a 1:1 ratio. A 5 ml pulse of the feed was contacted with the 100 ml column of resin, followed by 1.75 BV of 70% hexane/30% ethanol. The column was run at ambient temperature and a flow rate of 2.5 mls/minute. The results indicate a good separation of PC from the PE and PI, as well as a separation of the PC from sterols. The effluent history of this Example is shown in Figure 7.

[0071] Example 7.

[0072] 330 mls of a Kowa R2000P (Bell Pearl) phenolic resin was slurried in 70% hexane/30% ethanol and loaded in a 25 mm glass column. The column was washed with 3 BV of 70% hexane/30% ethanol. A feed was prepared by mixing 10 parts soy solubles (60% dry solids), 5 parts hexane, 2.5 parts ethanol, and 2 parts deionized water. 10 ml of the feed was contacted with the resin, followed by 1.5 BV of 70% hexane/30% ethanol. The column was run at ambient temperature and a flow rate of 10 mls/minute. The results indicate a good separation of PC from PE and PI, and the effluent history is shown in Figure 8.

[0073] Example 8.

[0074] The separation and purification of PC with non-ionic adsorbents was evaluated. In this Example, soy miscella was used as feed. 400 mls of the Mitsubishi SP850 resin (a highly porous, styrenic adsorbent resin) was loaded into a 25 mm glass column and the resin was washed with 3 BV of 60% ethanol in water, followed by washing with 3 BV of 100% ethanol. The column was also rinsed with 60% ethanol in water. 13 BV of filtered soy miscella (about 6% dry solids) was passed through the resin column heated at 44°C and the raffinate was collected. After running the soy miscella over the column, the heat was removed from the column and the rest of the Example was done at ambient temperature.

[0075] 1 BV of 60% ethanol in water was passed through the resin column producing an effluent referred to as the rinse fraction. The resin column was eluted with 2 BV of ethanol and the collected product was referred to as the ethanol product. The resin column was contacted with 2BV of 80% hexane in ethanol producing an effluent referred to as the hexane product. The resin column was then rinsed with 1BV of 60% ethanol in water and the effluent was collected

and referred to as the reset. The various effluents that were collected were analyzed for PA, PE, PC, and PI contents and the results are shown in Table 3.

[0076] Table 3.

					PE + PC + PI		
Sample ID	PA	PE	PC	PI	Total	Total %	%PC
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg		
Rinse fraction	121	347	1219	84	1650	0.1650	74
Raffinate	<LOD	<LOD	<LOD	21	21	0.0021	0
Ethanol product	329	258	2829	421	3499	0.3499	81
Hexane product	1734	3373	10693	2516	16582	1.6582	64
Reset	659	597	502	1757	2856	0.2856	18

[0077] The results of this Example show good enrichment of PC as evidenced by the %PC as compared to the other phospholipids. The products obtained from this Example and 3 other similar phospholipid separations were de-solventized and analyzed. The resulting hexane product had a PC purity of 52.3% base on total solids, absolute purity, from feed miscella and an absolute purity of 3.1%. The results of such analysis are shown in Table 4.

[0078] Table 4.

	% solids	% PC solids basis	PC/PE ratio	% PLs solids basis	% others solids basis	PC yield, %
Soy miscella	6.26	3.1	2.95	5.4	94.6	-
Ethanol product	7.1	21.1	5.98	27.8	72.2	25*
Hexane product	75.7	52.3	2.62	91	9	75*

* indicates the averages of four different column runs

[0079] Example 9.

[0080] A 400 ml column of the Mitsubishi SP70 resin (a styrene-divinylbenzene resin) was set up using 50% hexane/50% ethanol as the eluent and soy miscella as the feed. Quarter bed volumes were collected and analyzed. The results are shown in Figure 9. The amount of PC appears to be the highest between the first and second bed volumes. A portion of the effluent between the first and second bed volumes was analyzed and the results are shown in Table 5.

[0081] Table 5.

Phospholipid analyzed	% phospholipid
PC	59.3
PE	7.8
PI	5.8
PA	8.4
Total phospholipid	81.3
Non-phospholipid components	18.7

[0082] Example 10.

[0083] 100 mls of a Mitsubishi SP70 resin was loaded into a 15 mm glass column and washed with 3 BV of 60% ethanol in water, followed by washing with 1 BV of 100% ethanol. The column was also rinsed with 1 BV of 60% ethanol in water. 10 BV of a feed comprising filtered soy miscella (about 6% dry solids) was passed through the column at about 5 mls/minute at ambient temperature and the raffinate was collected. 1.5 BV of 60% ethanol was passed through the column and the collection of effluent fractions was started. After the 60% ethanol, 1 BV of 100% ethanol was passed through the column, followed by 1.5 BV of 50% hexane/50% ethanol. The elution profile is shown in Figure 10.

[0084] Two 10 liter columns plumbed in series were set up and loaded with the Mitsubishi SP70 resin. The run conditions were substantially similar to those described above in reference to the 15 mm glass column. The products obtained from the two 10 liter columns, along with three other similar runs, were de-solventized and analyzed. The resulting hexane/ethanol product has a PC purity of 65% based on total solids. The results of the analysis are shown in Table 6.

[0085] Table 6.

Compound	Weight %	Compound	Weight %
PC	64.97	FFA	3.13
Lyso PC	0.57	Moisture	0.76
PE	9.18	Hydrolyzed amino acid	0.3
LPE	0.17	Free amino acid	0.02
APE	1.79	MAB	0.06
PI	3.36	Phytates	< 20 ppm
PA	5.99	TAG	< 0.01
PG	1.8	Carbohydrates, below detection limit of 0.1	< 0.1
Total phospholipids*	87.83	Non-phospholipid components	4.27

*phospholipid composition was determined by 31P-NMR.

[0086] Example 11.

[0087] 100 mls of Mitsubishi SP70 resin were loaded into a 15 mm glass column, washed with 1 bed volume (BV) of specially denatured alcohol (SDA) 3C (formula of the denaturant), 190 proof (ethanol), followed by 1 BV of 65% hexane/35% SDA 3C, followed by 1 BV of SDA 3C, and followed by 1 BV of 75% SDA 3C. The feed was the supernatant from centrifuging soy miscella (6% dry solids) with the pH adjusted to 9 using 50% sodium hydroxide. 25 BV of the feed was passed through the resin in the column at 5 mls/minute at ambient temperature and the raffinate was collected. 1 BV of 75% ethanol was passed through the resin column, at which point the collection of the effluent fractions started. When the 75% SDA 3C rinse was complete, 1 BV of 100% SDA 3C, followed by 1.5 BV of 65% hexane/35% SDA 3C was passed through the column. The elution profile is shown in Figure 11.

[0088] Example 12.

[0089] 80 gram portions of soy miscella were pH adjusted across a range of pH's from 1 to 10 using 0.1 M HCl or 0.1 M NaOH and measured using a pH probe. In a 100 ml jar, 50 ml of each pH adjusted soy miscella portion (pH of 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10) was contacted with 10 grams of Mitsubishi SP70 resin and shaken occasionally at room temperature for 1 hour. After the hour, the liquid was decanted from each jar and sampled. To each jar, about 50 ml of 100% ethanol was added and the jar was allowed to stand for 20 minutes at room temperature with occasional mixing. After 20 minutes, the ethanol was decanted and sampled. The ethanol was replaced with hexane in the jars, and the jars were allowed to stand for 1 hour with occasional mixing. The hexane was decanted and sampled. All samples were analyzed for phospholipids. The resin which contacted the pH adjusted soy miscella whose pH was adjusted to 5 and 6 yielded the most PC, with the least amount of other phospholipids in the hexane washes.

[0090] Example 13.

[0091] Filtered soy miscella (having about 6% dry solids) was used as feed for this Example. The Kowa R2000P (Bell Pearl) phenolic resin was used in a carousel type simulated moving bed (SMB) system. The SMB used 12 100 ml resin columns and was configured in a 4-3-4-1 configuration as shown in Figure 12. These conditions resulted in the system performance shown in Table 7.

[0092] Table 7.

Sample	PA mg/kg	PE mg/kg	PC mg/kg	PI mg/kg	Volume ml	Mass grams	PC purity %	PC Yield %

SMB Feed	5685	6999	21979	7534	-	-	52.09	-
SMB Product	0	28	708	0	420	296.9	96.16	84.65
SMB Raffinate	604	609	300	827	180	127.3	-	-

[0093] The best results from this Example were obtained with the following conditions. Step Time: 5.6 minutes. Feed Rate: 1.0 mls/minute. Enrichment Rate: 7.0 mls/minute. Elution Rate: 17.1 mls/minute. Reload Rate: 2.9 mls/minute.

[0094] The ratios of PC to the other phospholipids indicate the degree of separation achieved with the Kowa resin in a continuous SMB operation. The feed had a PC ratio to the other three phospholipids of approximately 3:1, while the product has 25:1 (PC/PE ratio) to over 100:1 (PC/PA, PC/PI ratios) PC ratio. These ratios are shown in Table 8.

[0095] Table 8.

Sample	PC/PA Ratio	PC/PE Ratio	PC/PI Ratio
SMB Feed	3.87	3.14	2.92
SMB Product	>100	25.07	>100
SMB Raffinate	0.50	0.49	0.36

[0096] Example 14.

[0097] 50 gram portions of crude soy lecithin were contacted with ethanol and pH adjusted across a range of 2 to 13 using 0.1 M HCl or 0.1 M NaOH. In a 250 mL jar, 100 mL of pH adjusted beverage grade ethanol was added to 50 g each of the crude lecithin and shaken occasionally at room temperature for 10 minutes. After settling for several minutes, the light liquid phase was decanted from each jar and sampled. After settling, the heavy residue phase was also sampled. The extraction results ranges from 18% to 26% where the best results appeared to be obtained (in terms of %PC extracted and a ratio of PC/PA) at an ethanol pH of about 13.2. Conditions of the reactions and the results are presented in Tables 9 and 10.

[0098] Table 9.

Sample ID		PA (g)	%	PE (g)	%	PC (g)	%	PI(g)	%	l-PC (g)	%	Purity	PC/PE	PC/PA
67-B1	lecithin	2.838		8.187		8.879		3.712		0.315		37.6%	1.08	3.13
67-E1	pH?	0.140	4.9%	0.487	5.9%	1.585	17.8%	0.059	1.58%	0.124	39.31%	69.8%	3.26	11.34
67-R1	pH?	2.913		7.581		7.082		3.972		0.253		32.9%	0.93	2.43
		108%		99%		98%		109%		120%				
67-B2	lecithin	2.854		8.235		8.930		3.734		0.316		37.6%	1.08	3.13
67-E2	pH=13.21	0.161	5.6%	0.544	6.6%	1.620	18.1%	0.073	1.96%	0.122	38.43%	67.6%	2.98	10.06
67-R2	pH=13.21	2.737		6.753		6.236		3.810		0.216		31.9%	0.92	2.28
		102%		89%		88%		104%		107%				
67-B3	lecithin	2.853		8.231		8.927		3.732		0.316		37.6%	1.08	3.13
67-E3	pH=13.12	0.235	8.2%	0.698	8.5%	2.419	27.1%	0.044	1.18%	0.122	38.43%	71.2%	3.46	10.30
67-R3	pH=13.12	3.057		7.469		6.881		3.896		0.235		32.3%	0.92	2.25
		115%		99%		104%		106%		113%				
67-B4	lecithin	2.857		8.244		8.941		3.738		0.317		37.6%	1.08	3.13
67-E4	pH=12.76	0.183	6.4%	0.490	5.9%	1.684	18.8%	0.000	0.00%	0.112	35.25%	69.7%	3.44	9.20
67-R4	pH=12.76	2.709		6.815		6.830		0.000		0.221		34.7%	1.00	2.52
		101%		89%		95%		0%		105%				
67-B7	lecithin	2.843		8.204		8.897		3.720		0.315		37.6%	1.08	3.13
67-E7	pH=6.7 (n	0.273	9.6%	0.739	9.0%	2.332	26.2%	0.042	1.12%	0.103	32.53%	68.9%	3.16	8.55
67-R7	pH=6.7 (n	2.723		6.888		6.687		3.659		0.253		33.5%	0.97	2.46
		105%		93%		101%		99%		113%				
67-B6	lecithin	2.869		8.277		8.976		3.753		0.318		37.6%	1.08	3.13
67-E6	pH=3.83	0.204	7.1%	0.515	6.2%	1.999	22.3%	0.053	1.42%	0.132	41.46%	72.1%	3.88	9.79
67-R6	pH=3.83	2.690		7.530		6.840		3.516		0.207		33.2%	0.91	2.54
		101%		97%		98%		95%		107%				
67-B5	lecithin	2.837		8.186		8.877		3.711		0.315		37.6%	1.08	3.13
67-E5	pH=2.85	0.202	7.1%	0.514	6.3%	2.042	23.0%	0.051	1.37%	0.109	34.75%	72.7%	3.97	10.11
67-R5	pH=2.85	2.633		6.790		5.596		3.513		0.183		30.2%	0.82	2.13
		100%		89%		86%		96%		93%				

Sample ID		PA (g)	%	PE (g)	%	PC (g)	%	PI(g)	%	I-PC (g)	%	Purity	PC/PE	PC/PA
67-B1	lecithin	2.838		8.187		8.879		3.712		0.315		37.6%	1.08	3.13
67-E1	pH?	0.140	4.9%	0.487	5.9%	1.585	17.8%	0.059	1.58%	0.124	39.31%	69.8%	3.26	11.34
67-R1	pH?	2.913		7.581		7.082		3.972		0.253		32.9%	0.93	2.43
		108%		99%		98%		109%		120%				
67-B2	lecithin	2.854		8.235		8.930		3.734		0.316		37.6%	1.08	3.13
67-E2	pH=13.21	0.161	5.6%	0.544	6.6%	1.620	18.1%	0.073	1.96%	0.122	38.43%	67.6%	2.98	10.06
67-R2	pH=13.21	2.737		6.753		6.236		3.810		0.216		31.9%	0.92	2.28
		102%		89%		88%		104%		107%				
67-B3	lecithin	2.853		8.231		8.927		3.732		0.316		37.6%	1.08	3.13
67-E3	pH=13.12	0.235	8.2%	0.698	8.5%	2.419	27.1%	0.044	1.18%	0.122	38.43%	71.2%	3.46	10.30
67-R3	pH=13.12	3.057		7.469		6.881		3.896		0.235		32.3%	0.92	2.25
		115%		99%		104%		106%		113%				
67-B4	lecithin	2.857		8.244		8.941		3.738		0.317		37.6%	1.08	3.13
67-E4	pH=12.76	0.183	6.4%	0.490	5.9%	1.684	18.8%	0.000	0.00%	0.112	35.25%	69.7%	3.44	9.20
67-R4	pH=12.76	2.709		6.815		6.830		0.000		0.221		34.7%	1.00	2.52
		101%		89%		95%		0%		105%				
67-B7	lecithin	2.843		8.204		8.897		3.720		0.315		37.6%	1.08	3.13
67-E7	pH=6.7 (n	0.273	9.6%	0.739	9.0%	2.332	26.2%	0.042	1.12%	0.103	32.53%	68.9%	3.16	8.55
67-R7	pH=6.7 (n	2.723		6.888		6.687		3.659		0.253		33.5%	0.97	2.46
		105%		93%		101%		99%		113%				
67-B6	lecithin	2.869		8.277		8.976		3.753		0.318		37.6%	1.08	3.13
67-E6	pH=3.83	0.204	7.1%	0.515	6.2%	1.999	22.3%	0.053	1.42%	0.132	41.46%	72.1%	3.88	9.79
67-R6	pH=3.83	2.690		7.530		6.840		3.516		0.207		33.2%	0.91	2.54
		101%		97%		98%		95%		107%				
67-B5	lecithin	2.837		8.186		8.877		3.711		0.315		37.6%	1.08	3.13
67-E5	pH=2.85	0.202	7.1%	0.514	6.3%	2.042	23.0%	0.051	1.37%	0.109	34.75%	72.7%	3.97	10.11
67-R5	pH=2.85	2.633		6.790		5.596		3.513		0.183		30.2%	0.82	2.13
		100%		89%		86%		96%		93%				

[0099] Table 10.

Sample ID		PA (g)	%	PE (g)	%	PC (g)	%	PI(g)	%	I-PC (g)	%	Purity	PC/PE	PC/PA
Feed	lecithin	2.838		8.187		8.879		3.712		0.315		37.6%	1.08	3.13
Light Phase	pH?	0.140	4.9%	0.487	5.9%	1.585	17.8%	0.059	1.58%	0.124	39.31%	69.8%	3.26	11.34
Heavy Phase	pH?	2.913		7.581		7.082		3.972		0.253		32.9%	0.93	2.43
Amount accounted for		108%		99%		98%		109%		120%				
Feed	lecithin	2.854		8.235		8.930		3.734		0.316		37.6%	1.08	3.13
Light Phase	pH=13.21	0.161	5.6%	0.544	6.6%	1.620	18.1%	0.073	1.96%	0.122	38.43%	67.6%	2.98	10.06
Heavy Phase	pH=13.21	2.737		6.753		6.236		3.810		0.216		31.9%	0.92	2.28
Amount accounted for		102%		89%		88%		104%		107%				
Feed	lecithin	2.853		8.231		8.927		3.732		0.316		37.6%	1.08	3.13
Light Phase	pH=13.12	0.235	8.2%	0.698	8.5%	2.419	27.1%	0.044	1.18%	0.122	38.43%	71.2%	3.46	10.30
Heavy Phase	pH=13.12	3.057		7.469		6.881		3.896		0.235		32.3%	0.92	2.25
Amount accounted for		115%		99%		104%		106%		113%				
Feed	lecithin	2.857		8.244		8.941		3.738		0.317		37.6%	1.08	3.13
Light Phase	pH=12.76	0.183	6.4%	0.490	5.9%	1.684	18.8%	0.000	0.00%	0.112	35.25%	69.7%	3.44	9.20
Heavy Phase	pH=12.76	2.709		6.815		6.830		0.000		0.221		34.7%	1.00	2.52
Amount accounted for		101%		89%		95%		0%		105%				
Feed	lecithin	2.843		8.204		8.897		3.720		0.315		37.6%	1.08	3.13
Light Phase	pH=6.7 (as is)	0.273	9.6%	0.739	9.0%	2.332	26.2%	0.042	1.12%	0.103	32.53%	68.9%	3.16	8.55
Heavy Phase	pH=6.7 (as is)	2.723		6.888		6.587		3.659		0.253		33.5%	0.97	2.46
Amount accounted for		105%		93%		101%		99%		113%				
Feed	lecithin	2.837		8.186		8.877		3.711		0.315		37.6%	1.08	3.13
Light Phase	pH=2.85	0.202	7.1%	0.514	6.3%	2.042	23.0%	0.051	1.37%	0.109	34.75%	72.7%	3.97	10.11
Heavy Phase	pH=2.85	2.633		6.790		5.596		3.513		0.183		30.2%	0.82	2.13
Amount accounted for		100%		89%		86%		96%		93%				
Feed	lecithin	2.869		8.277		8.976		3.753		0.318		37.6%	1.08	3.13
Light Phase	pH=3.83	0.204	7.1%	0.515	6.2%	1.999	22.3%	0.053	1.42%	0.132	41.46%	72.1%	3.88	9.79
Heavy Phase	pH=3.83	2.690		7.530		6.840		3.516		0.207		33.2%	0.91	2.54
Amount accounted for		101%		97%		98%		95%		107%				

[00100] Example 15.

[00101] pH adjusted portions of crude (unfiltered) soy lecithin were contacted with wet denatured ethanol. The extraction was performed similar to Example 14 to obtain a light phase and a heavy phase. In this Example, 50 mL of the light phase was used as the feed (the pH adjusted crude lecithin in wet denatured ethanol) and was loaded at 5 mL/min. and ambient temperature onto a 15 mm glass column containing 100 mL of Mitsubishi SP 70 brand resin in wet denatured ethanol. The eluent was collected as raffinate 1. 1 BV of 60% wet denatured ethanol in water was run and the eluent was collected as raffinate 2. Once the 60% wet denatured ethanol rinse was completed, 1 BV of wet denatured ethanol, followed by a 1.5 BV of 80:20 hexane: wet denatured ethanol mixture was passed through the column to waste. The elution profile is shown in the graph of Figure 14. The PC/PA ratio from 1.5 to 2.0 BV was greater than 20.

[00102] Example 16.

[00103] A C-SEP system was set up, loaded with Mitsubishi SP70 resin, configured in a 1-1-2-1-1-4 configuration as shown in Figure 14. Soy miscella feed was pH adjusted to 9 using 50% NaOH, followed by settling. The liquid portion of this soy miscella (having about 6% dry solids) was used as feed for this Example. The conditions resulted in the system performance shown in Table 11.

[00104] Table 11.

Sample	PA mg/kg	PE mg/kg	PC mg/kg	PI mg/kg	PC/PA Ratio	Mass grams	PC purity % S. B.	PC Yield %
SMB Feed	299	430	1473	467	4.92	4005	3.12	-
SMB Prod 1	612	2956	18789	924	30.68	268	69.54	85.38
SMB Prod 2	803	1880	948	1201	1.18	260	15.39	-

[00105] Example 17.

[00106] 100 mls of Dow XAD18 resin were loaded into a 15 mm glass column, washed with 1BV of SDA 3C, 190 proof (ethanol), followed by 1 BV of 60% SDA 3C. 15 BV of the feed was passed through the resin in the column at 5 mls/minute at 40°C, followed by 1 BV of 60 SDA 3C. The column was eluted with 3 BV of 100% SDA 3C and 0.5 BV fractions were collected. The elution was completed with 2 BV of 60% hexane/40% SDA 3C and 1 BV fractions were collected. The elution profile is shown in Figure 15. 85% of the PC elutes with the SDA 3C with the PC/PA ratio being 25.2.

[00107] Example 18.

[00108] 58 kilograms of PC C-SEP product at approximately 2% - 2.5% solids were collected into five 5 gallon partially filled buckets. Collectively, the buckets contained 967.3 grams of phospholipids, 733.6 grams of which were PC. A precipitate forms in the product upon exiting the C-SEP. The amount of the precipitate is small and is 60% phospholipids, however only 0.33% of the precipitate is PC. The liquid portion PC C-SEP product is then concentrated to approximately 10% solids and collected into a single 5 gallon bucket. Upon sitting at least 24 hours, a second precipitate forms. The second precipitate which accounts for about 1% of the mass is 75% phospholipids with 12% of the precipitate being PC. The liquid portion is then concentrated to approximately 50% solids. The 50% solids concentrate weighs 1.7 kg and contains 784.1 grams of phospholipids of which 656.4 grams are PC. No further precipitation is

observed. Removing the precipitates in stages can be used to enhance the overall PC purity of the final product. Table 12 illustrates the purity enhancement. PC purity on a phospholipid basis is PC/PC+PA+PE+PI.

[00109] Table 12.

	PC/PA	PC/PE	PC purity on a phospholipid basis
PC CSEP PD (2% solids)	15.9	5.7	75.84
First Evap PD (10% solids	21.5	6.1	81.05
Second Evap PD (50% solids)	25.5	7.9	83.72

[00110] Example 19.

[00111] In this example, the chromatographic system using Mitsubishi SP70 Non-ionic resin is configured in a 2-2-4-2-2-8 sequence in a simulated moving bed (SMB) apparatus. There is twenty (20) 100 mL columns, for a total resin volume of 2.0 liters. Zone I is the adsorption zone; zone II is the Feed Rinse zone; zone III is the Solvent Rinse Zone, zone IV is the Enrichment Zone; zone V is the Solvent Stripping zone, and zone VI is the Reload zone (Figure 16). The SMB apparatus contains 20 columns on a carousel, and provisions for rotating the columns in the direction opposite the flow of fluid at defined intervals, called the "Step Time". The step time is 20 minutes.

[00112] Zone I (the Adsorption Zone) is defined by the feed (Soy Miscella) inlet ports, all plumbed to run in parallel; and there is 8 columns in this zone (columns 13-20 in Figure 16). Soy miscella feed containing ~0.1 -0.2% PC is applied continuously in this zone; where, the phospholipids are preferentially adsorbed onto the resin, while most of the other solids in stream continue through the resin bed and are discharged in the raffinate port.

[00113] Zone II (the Feed Rinse zone) is columns 11 & 12 in Figure 16. The solvent flow in this zone is a solution of 75% SDA-3C ethanol / 25% water (150 proof), and there is be two columns in this zone. The primary purpose of this zone is to ensure the removal of the feed impurities from the void fraction within the resin bed, and to rinse the resin.

[00114] Zone III (the Solvent Rinse zone) is columns 9 & 10 in Figure 16. There is two columns in this zone, as well. The primary purpose of this zone is to remove the bulk of the water from the mobile phase, using 190 proof SDA-3C ethanol, prior to the hexane elution zone.

[00115] Zone IV (the Enrichment Zone) is the zone defined by the product 2 discharge and Enrichment inlet ports (columns 5 - 8 in Figure 16). The primary purpose of this zone is to separate the adsorbed phospholipids to enhance the purity of PC relative to the PE, PI, and PA.

[00116] Zone V (Solvent Stripping zone) is the zone defined by the 65 / 35 Hexane / Ethanol rinse inlet and the Product 2 (P2) outlet (Columns 3 & 4 in Figure 16). The purpose of this zone is to strip the remaining phospholipids off the resin to prepare it for adsorption.

[00117] Zone VI (the Reload zone) is columns 1 & 2 in Figure 16. The purpose of this zone is to remove the hexane from the resin bed prior to returning the columns to the adsorption zone. This is important because hexane in the resin bed will cause a decrease in adsorption of phospholipids, when the column rotates into the adsorption zone.

[00118] Example 20. 2-Step Chromatography for High Purity PC.

[00119] In this example, the process involves a two-step chromatographic separation to produce a product of >90% PC purity. Between the two steps there is a solvent removal step and a solvent addition step prior to the second stage chromatographic system. The first stage chromatographic system can be either the styrene based resin (such as SP70) using miscella / molasses / light phase from ethanol extracted lecithin (EEL) as feed; or the phenolic based resin (such as XAD761) using the light phase of hexane extracted lecithin (HEL) as feed. The primary product from any of these processes can be further processed using a further embodiment of the process. In this case the product is exposed to further purification as described in Figure 18 above. The specifics for this processing are detailed herein.

[00120] For the product from any of these processes as feed, the adsorbent could be either the phenolic based resin or the styrene based resin. In the event that the phenolic based resin (XAD761, Bellpearl, or similar) is used, the required feed solvent (Figure 17) is hexane. The solvents required in the chromatographic operation are a hexane rinse with ethanol elution (or a combination of ethanol and hexane), followed by a hexane rinse. If the non-ionic styrene based resin (SP70) resin is used, the required feed solvent (Figure 17) would be ethanol and water (to approximately 60% ethanol / 35% water). The solvents required in the chromatographic operation are the ethanol/water rinse, followed by ethanol rinse; followed by hexane / ethanol elution, followed by an ethanol rinse.

[00121] The present invention has been described with reference to certain exemplary and illustrative embodiments, compositions and uses thereof. However, it will be recognized by persons having ordinary skill in the art that various substitutions, modifications or combinations of any of the exemplary embodiments may be made without departing from the scope of the invention. Thus, the invention is not limited by the description of the exemplary and illustrative embodiments, but rather by the appended claims.

CLAIMS

What is claimed is:

1. A process of fractionating phospholipids, the process comprising:

placing a phospholipid containing material in contact with an adsorbent, such that the adsorbent associates with at least one phospholipid of the phospholipid containing material.

2. The process according to claim 1, wherein the adsorbent is a polymeric resin.

3. The process according to claim 2, wherein the polymeric resin is selected from the group consisting of a phenolic resin, a styrene resin, a non-ionic resin, an acrylic resin, a cation-exchange resin, a weak-base resin, and combinations of any thereof.

4. The process according to claim 2, wherein the polymeric resin is a styrene resin or a phenolic resin.

5. The process according to any one of claims 1-4, wherein the phospholipid containing material is selected from the group consisting of miscella, molasses, lecithin, and combinations of any thereof.

6. The process according any one of claims 1-5, wherein the phospholipid containing material is miscella.

7. The process according to any one of claims 1-6, further comprising eluting the at least one phospholipid from the adsorbent.

8. The process according to claim 7, wherein eluting the at least one phospholipid from the adsorbent comprises contacting an elution solvent with the adsorbent.

9. The process according to claim 7 or claim 8, wherein the phospholipid containing material contains less than 5% by weight of phospholipids and the at least one phospholipid eluted from the adsorbent contains more than 50% by weight of the phospholipids.

10. The process according to any one of claims 7-9, further comprising placing the at least one phospholipid eluted from the adsorbent in contact with a second adsorbent.

11. The process according to claim 10, further comprising mixing the at least one phospholipid eluted from the adsorbent with a solvent before placing in contact with the second absorbent.

12. The process according to any one of claims 8-11, further comprising collecting the eluted, at least one phospholipid.

13. The process according to any one of claims 1-12, further comprising:

mixing a solvent with the phospholipid containing material, thus producing a feed; and

wherein placing the phospholipid containing material in contact with the adsorbent comprises placing the feed in contact with the adsorbent.

14. The process according to claim 13, further comprising eluting the at least one phospholipid from the adsorbent with an elution solvent.

15. The process according to claim 14, further comprising collecting the eluted, at least one phospholipid and the elution solvent.

16. The process according to claim 15, further comprising allowing a precipitate to form in the eluted, at least one phospholipid and the elution solvent.

17. The process according to claim 16, further comprising:

removing the precipitate from the eluted, at least one phospholipid and the elution solvent; and

concentrating the at least one phospholipid in the elution solvent.

18. The process according to any one of claims 11-17, wherein the solvent is selected from the group consisting of an alcohol, an alkane, water, and combinations of any thereof.

19. The process according to any one of claims 1-18, wherein the at least one phospholipid is selected from the group consisting of phosphatidyl choline (PC), phosphatidyl ethanolamine (PE), phosphatidyl inositol (PI), phosphatidic acid (PA), and combinations of any thereof.

20. The process according to any one of claims 1-19, further comprising extracting the phospholipid containing material from a phospholipid source.

21. The process according to any one of claims 1-20, further comprising adjusting a pH of the phospholipid containing material to a pH of between 8.5-10.0.

22. The process according to any one of claims 1-21, further comprising removing solids from the phospholipid containing material.

23. The process according to any one of claims 1-22, wherein the adsorbent is located in a simulated moving bed chromatography system.

24. The process according to claim 18, wherein the solvent is the alkane and is selected from the group consisting of hexane, heptane, or a combination thereof.

25. The process according to any one of claims 1-26, wherein the phospholipids originate from a source selected from the group consisting of soybean, egg, canola, sunflower, corn, and combinations of any thereof.

26. A process of fractionating phospholipids, the process comprising:

mixing a phospholipid containing material selected from the group consisting of miscella, lecithin, molasses, and combinations of any thereof with a solvent, thus producing a feed;

placing the feed in contact in contact with a polymeric resin, such that the polymeric resin associates with at least one phospholipid of the phospholipid containing material; and

eluting the at least one phospholipid from the polymeric resin with an elution solvent.

27. The process of claim 26, wherein the polymeric resin is selected from the group consisting of a phenolic resin and a styrene resin.

28. The process of claim 26 or claim 27, wherein the solvent comprises an alcohol, an alkane, water, and combinations of any thereof.

29. The process of any one of claims 26-28, wherein the elution solvent comprises an alcohol, an alkane, water, and combinations of any thereof.

30. The process of any one of claims 26-29, wherein the phospholipid containing material is the miscella.

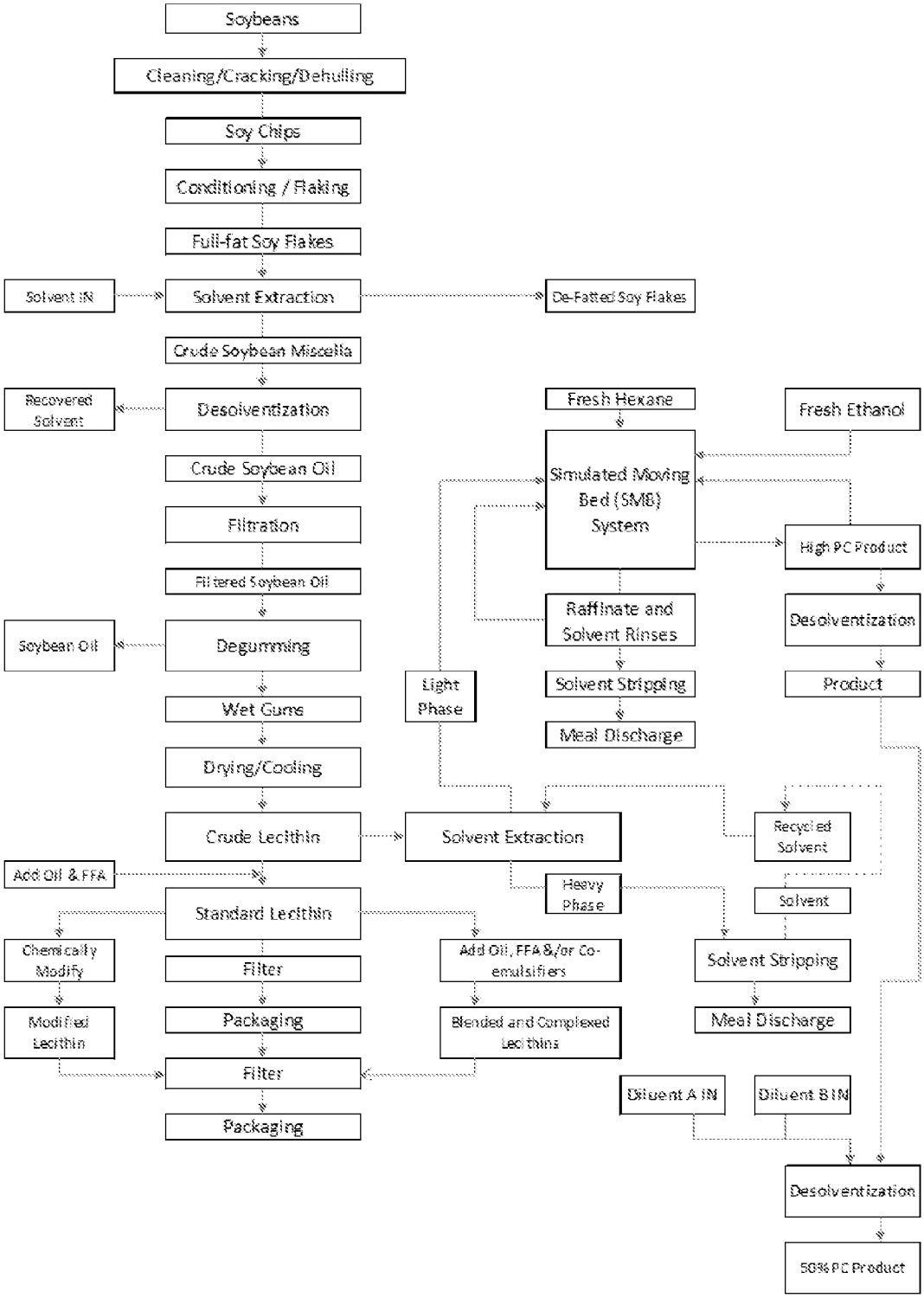
Soybeans
Cleaning/Cracking/Dehulling
Soy Chips
Conditioning / Flaking
Full-fat Soy Flakes
Solvent IN
Solvent Extraction
De-Fatted Soy Flakes
Crude Soybean Miscella
Recovered Solvent
Desolventization
Crude Soybean Oil
Filtration
Filtered Soybean Oil
Soybean Oil
Degumming
Wet Gums
Drying/Cooling
Crude Lecithin
Add Oil & FFA
Standard Lecithin
Chemically Modify
Filter
Modified Lecithin
Packaging
Filter
Packaging
Fresh Hexane
Fresh Ethanol
Simulated Moving Bed (SMB) System
High PC Product
Raffinate and Solvent Rinses
Desolventization
Light Phase
Solvent Stripping
Product
Meal Discharge
Solvent Extraction
Recycled Solvent
Heavy Phase
Solvent
Add Oil, FFA &/or Co-emulsifiers
Solvent Stripping
Blended and Complexed Lecithins
Meal Discharge
Diluent A IN
Diluent B IN
Desolventization
50% PC Product

FIGURE 1

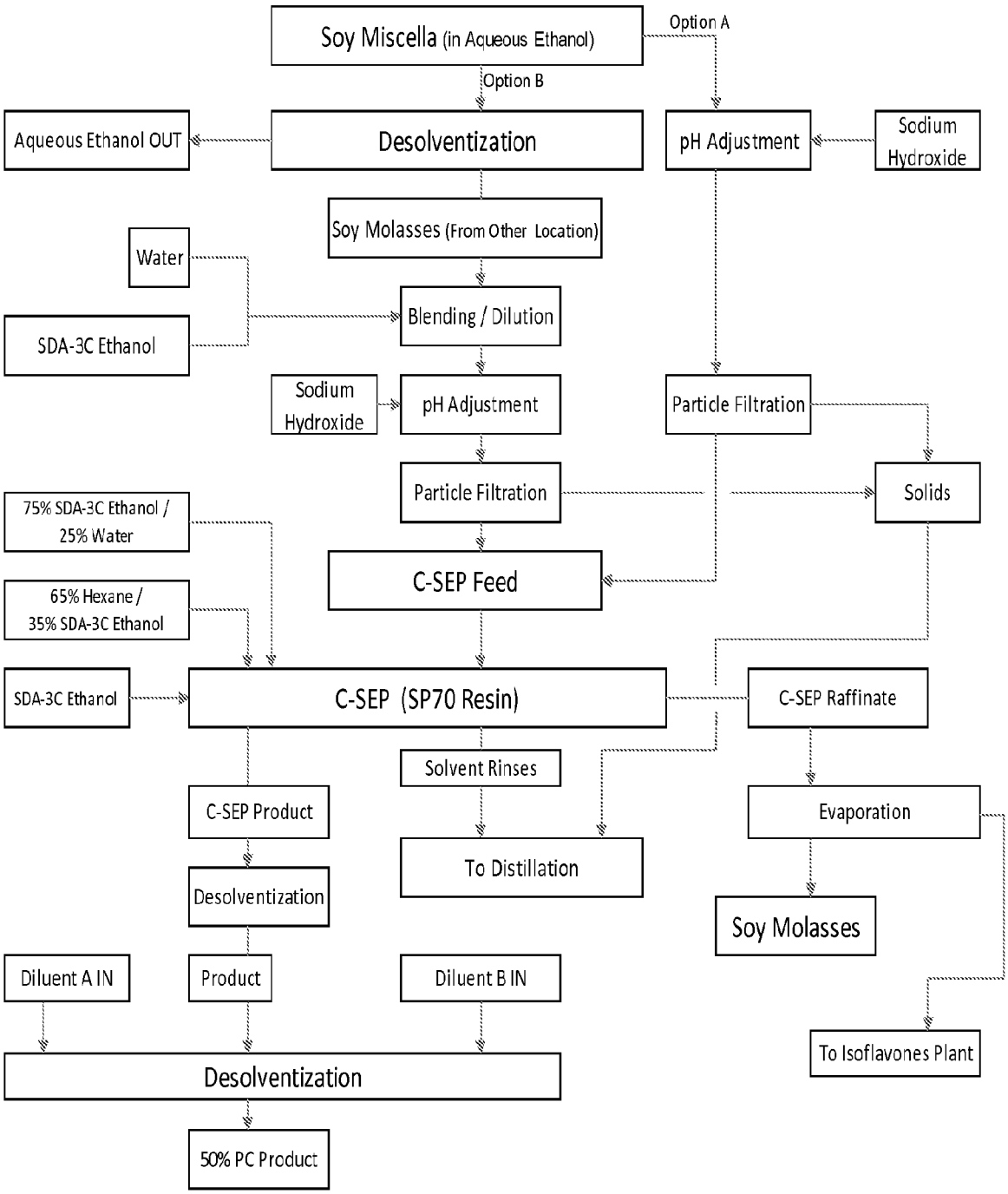
Soy Miscella (in Aqueous Ethanol)
Option A
Option B
Aqueous Ethanol OUT
Desolventization
pH Adjustment
Sodium Hydroxide
Soy Molasses (From Other Location)
Water
SDA-3C Ethanol
Blending / Dilution
Sodium Hydroxide
pH Adjustment
Particle Filtration
Particle Filtration
Solids
75% SDA-3C Ethanol / 25% Water
C-SEP Feed
65% Hexane / 35% SDA-3C Ethanol
SDA-3C Ethanol
C-SEP (SP70 Resin)
C-SEP Raffinate
Solvent Rinses
C-SEP Product
Evaporation
To Distillation
Desolventization
Soy Molasses
Diluent A IN
Product
Diluent B IN
To Isoflavones Plant
Desolventization
50% PC Product

FIGURE 2

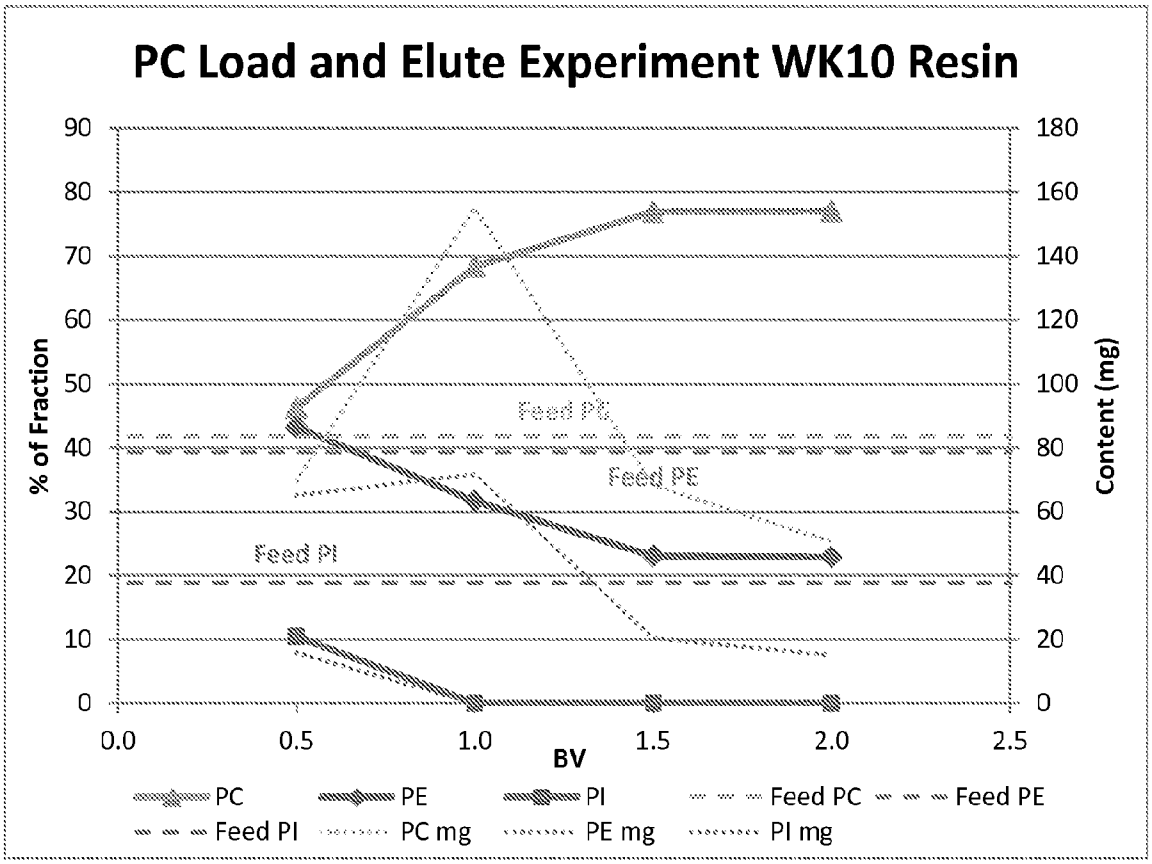
PC Load and Elute Experiment WK10 Resin
% of Fraction
Content (mg)
BV
Feed PC
Feed PE
Feed PI
PC
PE
PI
Feed PC
Feed PE
Feed PI
PC mg
PE mg
PI mg

FIGURE 3

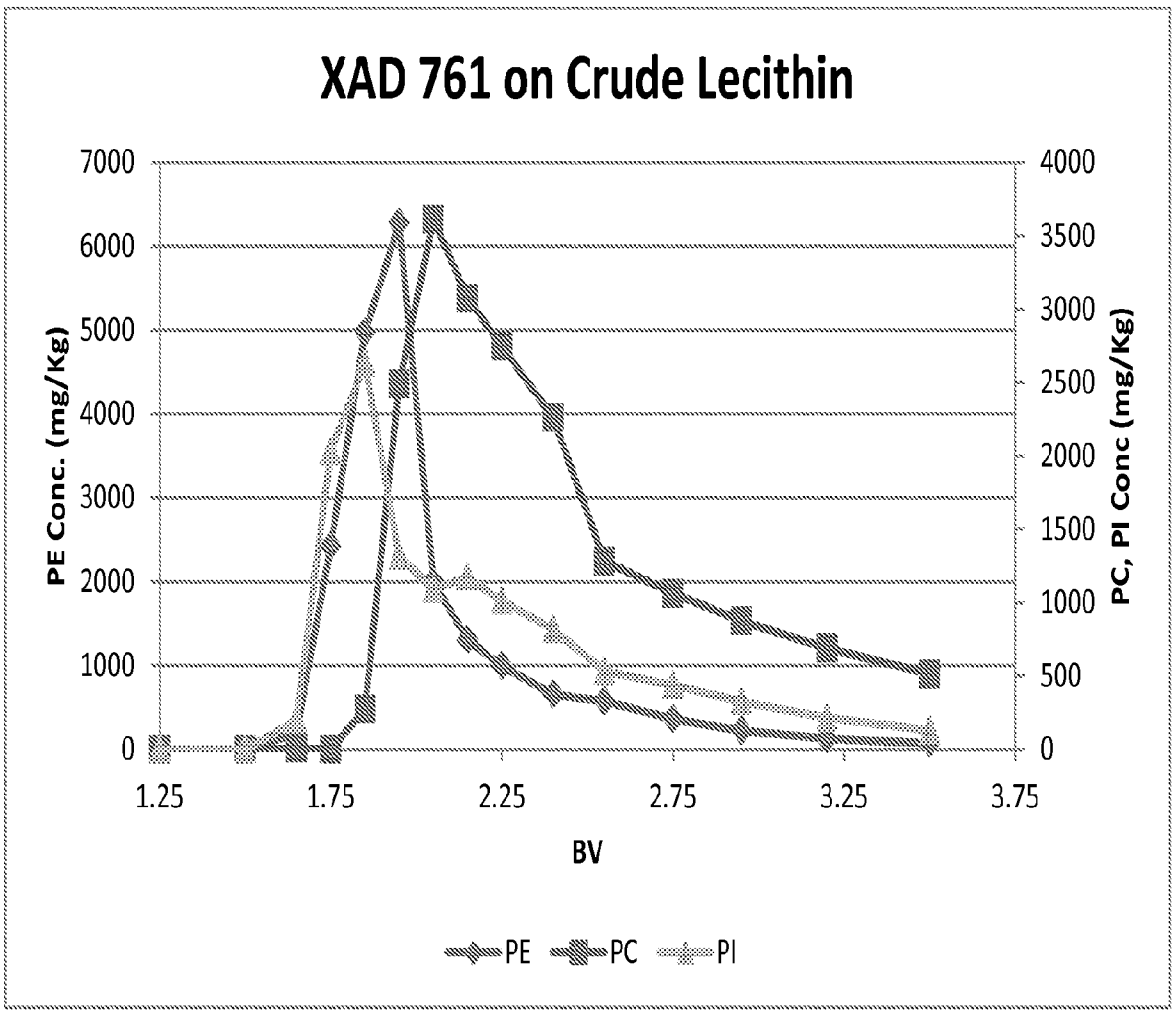
XAD 761 on Crude Lecithin
PE Conc. (mg/Kg)
PC, PI Conc (mg/Kg)
7000
6000
5000
4000
3000
2000
1000
0
4000
3500
3000
2500
2000
1500
1000
500
0
1.25
1.75
2.25
2.75
3.25
3.75
BV
PE
PC
PI

FIGURE 4

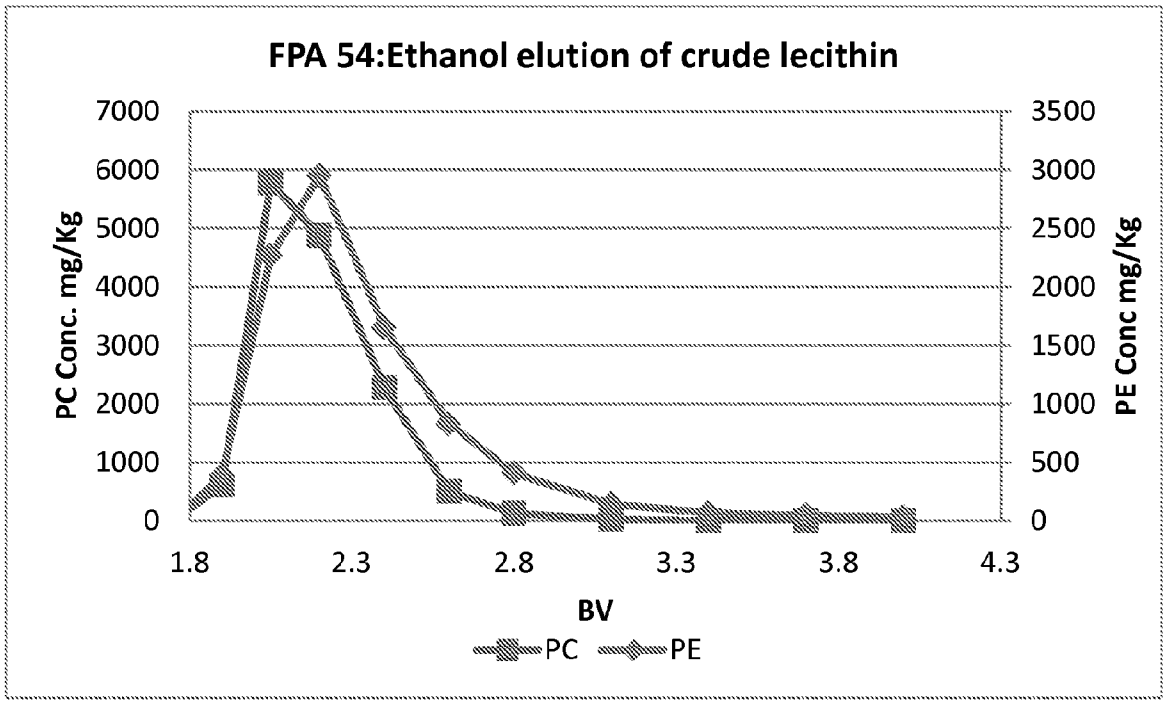
FPA 54:Ethanol elution of crude lecithin
PC Conc. mg/Kg
PE Conc mg/Kg
7000
6000
5000
4000
3000
2000
1000
0
3500
3000
2500
2000
1500
1000
500
0
1.8
2.3
2.8
3.3
3.8
4.3
BV
PC
PE

FIGURE 5

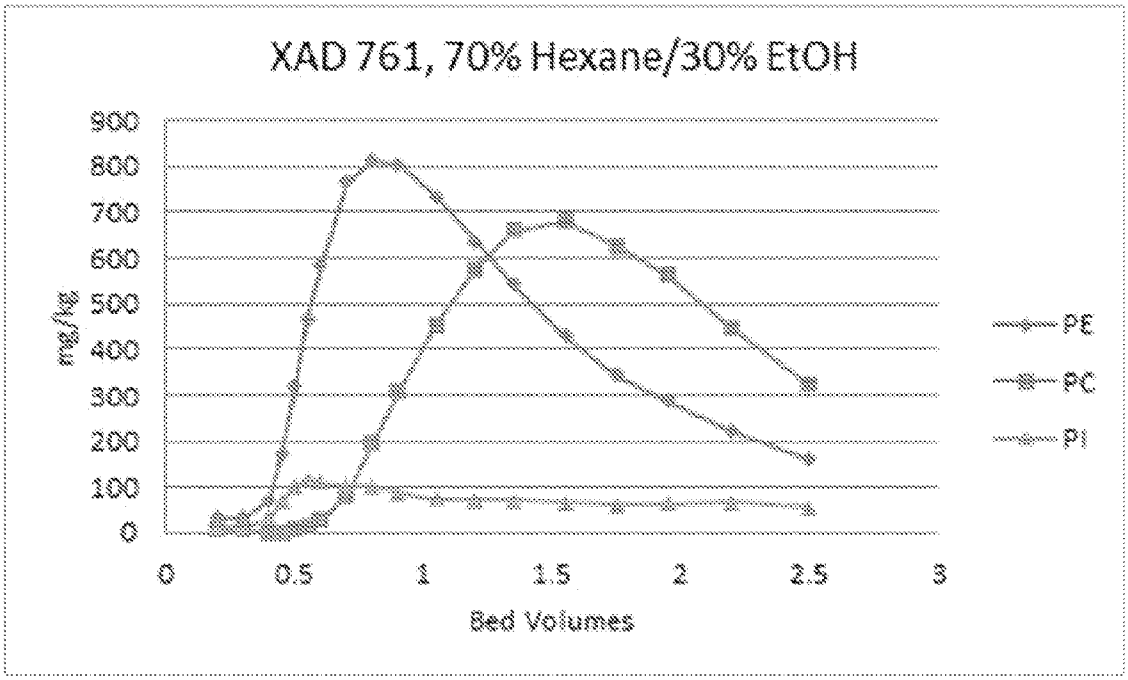
XAD 761, 70% Hexane/30% EtOH
900
800
700
600
500
400
300
200
100
0
mg/kg
0
0.5
1
1.5
2
2.5
3
Bed Volumes
PE
PC
PI

FIGURE 6

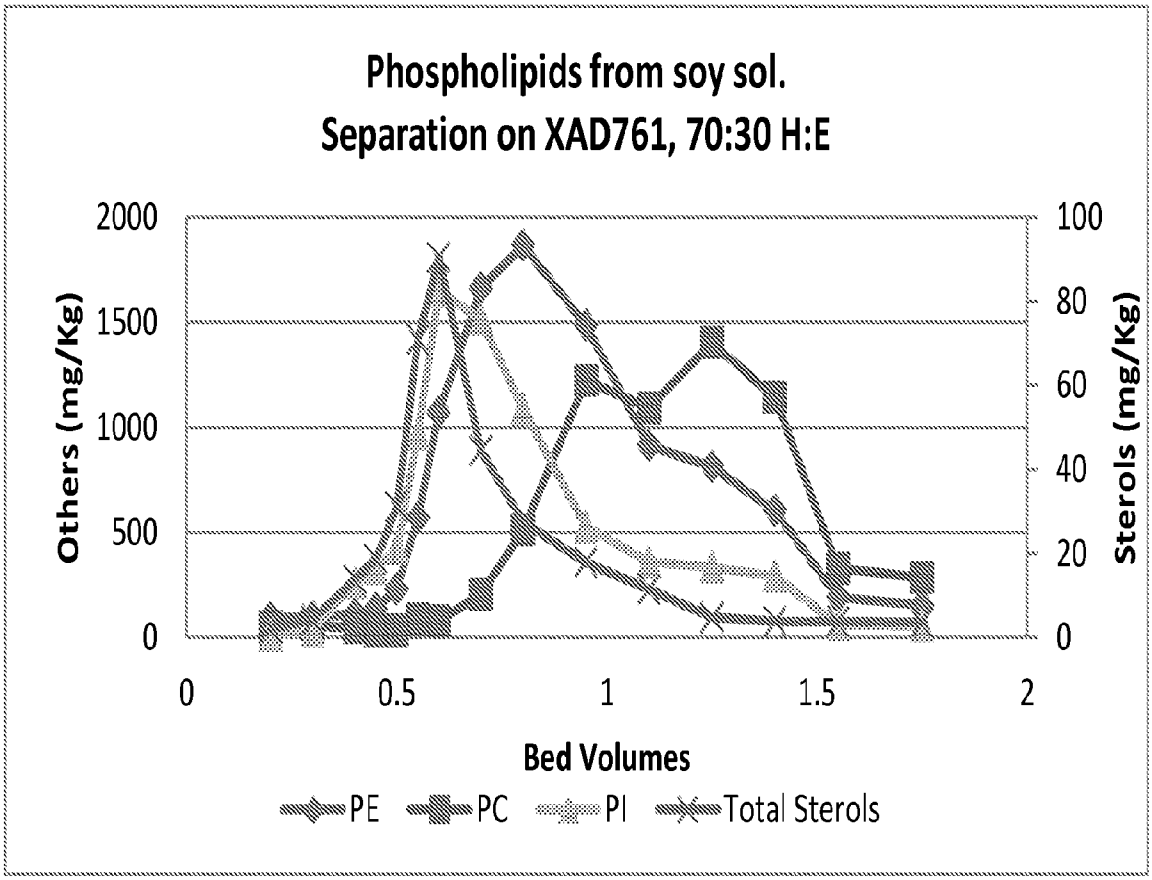
Phospholipids from soy sol.
Separation on XAD761, 70:30 H:E
Others (mg/Kg)
2000
1500
1000
500
0
Sterols (mg/Kg)
100
80
60
40
20
0
0
0.5
1
1.5
2
Bed Volumes
PE
PC
PI
Total Sterols

FIGURE 7

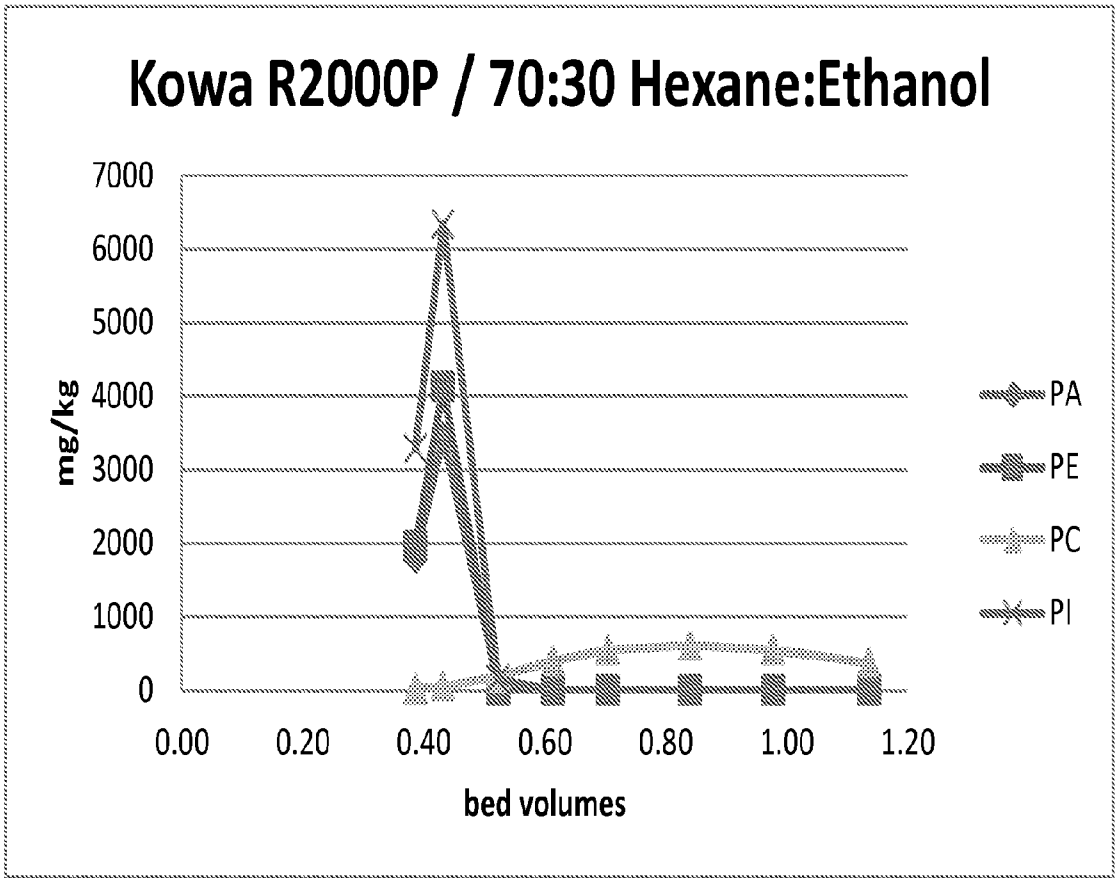
Kowa R2000P / 70:30 Hexane:Ethanol
7000
6000
5000
4000
3000
2000
1000
0
mg/kg
0.00
0.20
0.40
0.60
0.80
1.00
1.20
bed volumes
PA
PE
PC
PI

FIGURE 8

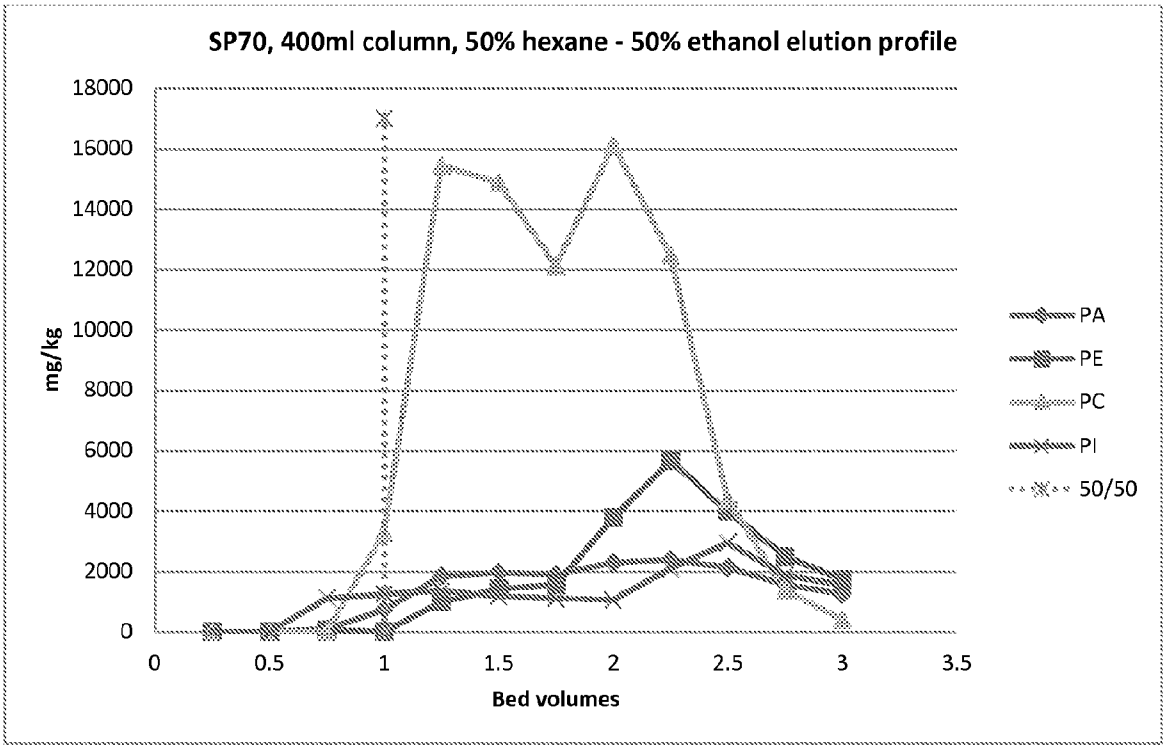
SP70, 400ml column, 50% hexane - 50% ethanol elution profile
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
mg/kg
0
0.5
1
1.5
2
2.5
3
3.5
Bed volumes
PA
PE
PC
PI
50/50

FIGURE 9

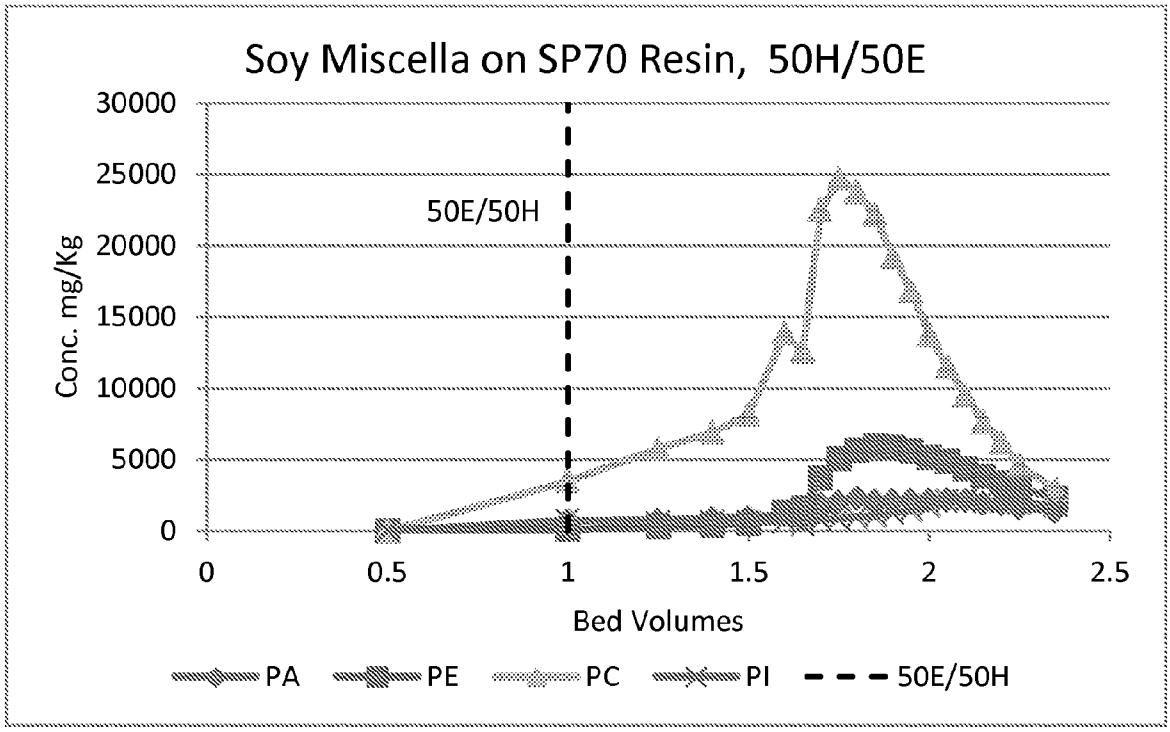
Soy Miscella on SP70 Resin, 50H/50E
30000
25000
20000
15000
10000
5000
0
Conc. mg/Kg
50E/50H
0
0.5
1
1.5
2
2.5
Bed Volumes
PA
PE
PC
PI
50E/50H

FIGURE 10

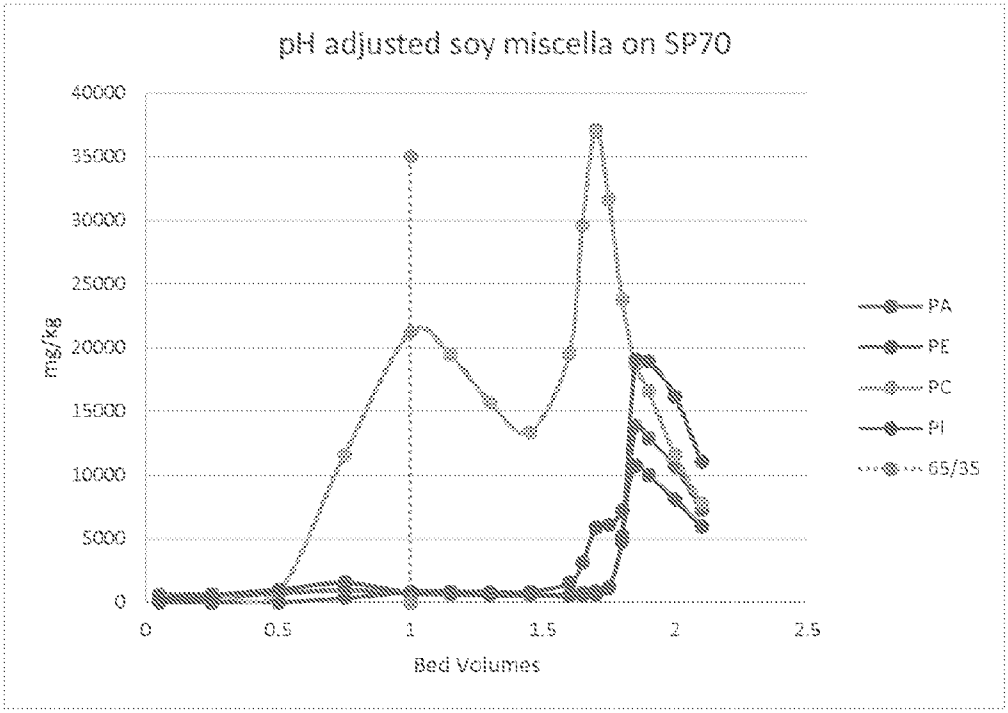
pH adjusted soy miscella on SP70
40000
35000
30000
25000
20000
15000
10000
5000
0
mg/kg
0
0.5
1
1.5
2
2.5
Bed Volumes
PA
PE
PC
PI
65/35

FIGURE 11

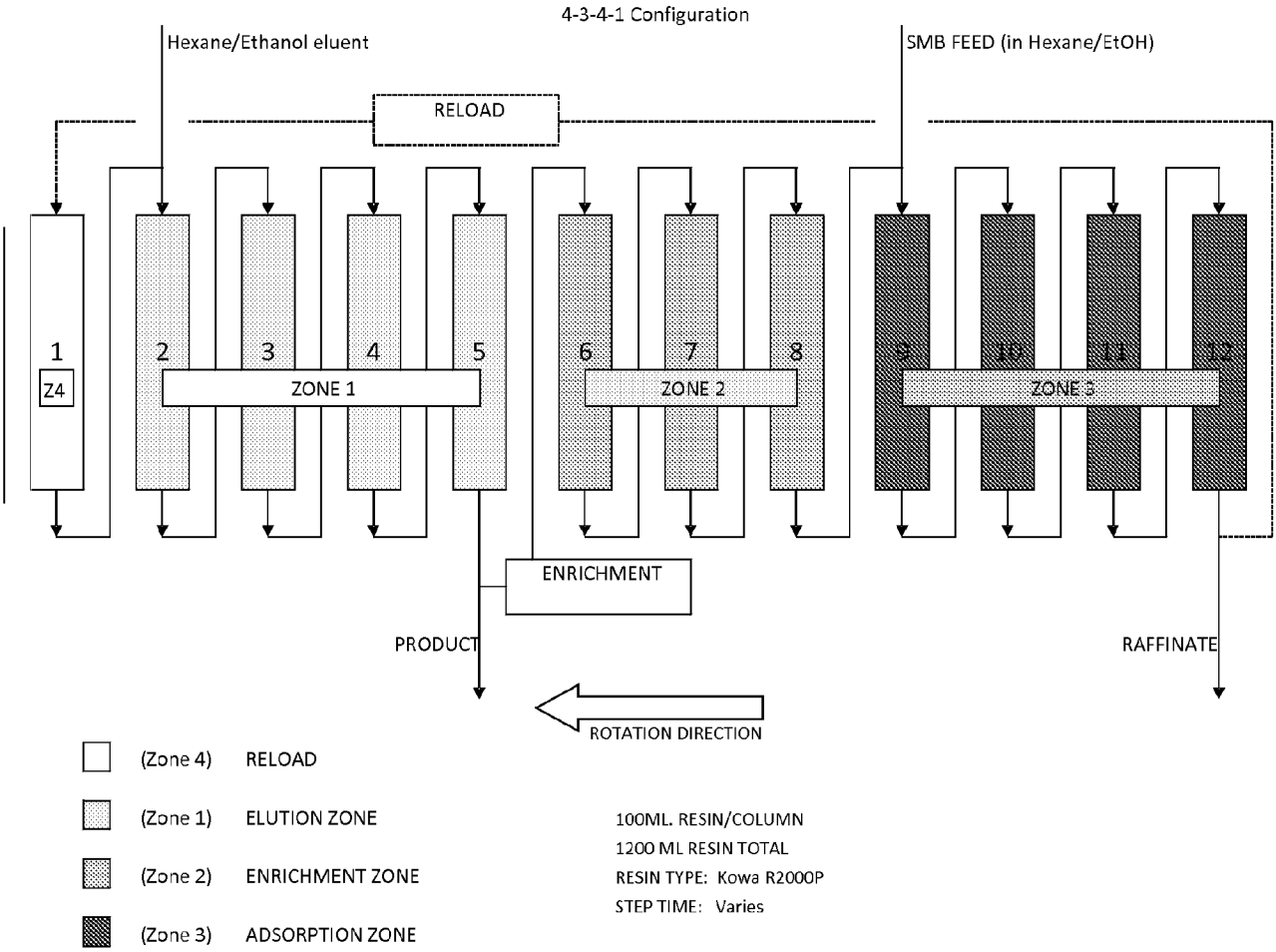
4-3-4-1 Configuration
Hexane/Ethanol eluent
SMB FEED (in Hexane/EtOH)
RELOAD
1
Z4
2
3
4
5
ZONE 1
6
7
8
ZONE 2
9
10
11
12
ZONE 3
ENRICHMENT
PRODUCT
RAFFINATE
ROTATION DIRECTION
(Zone 4) RELOAD
(Zone 1) ELUTION ZONE
(Zone 2) ENRICHMENT ZONE
(Zone 3) ADSORPTION ZONE
100ML. RESIN/COLUMN
1200 ML RESIN TOTAL
RESIN TYPE: Kowa R2000P
STEP TIME: Varies

FIGURE 12

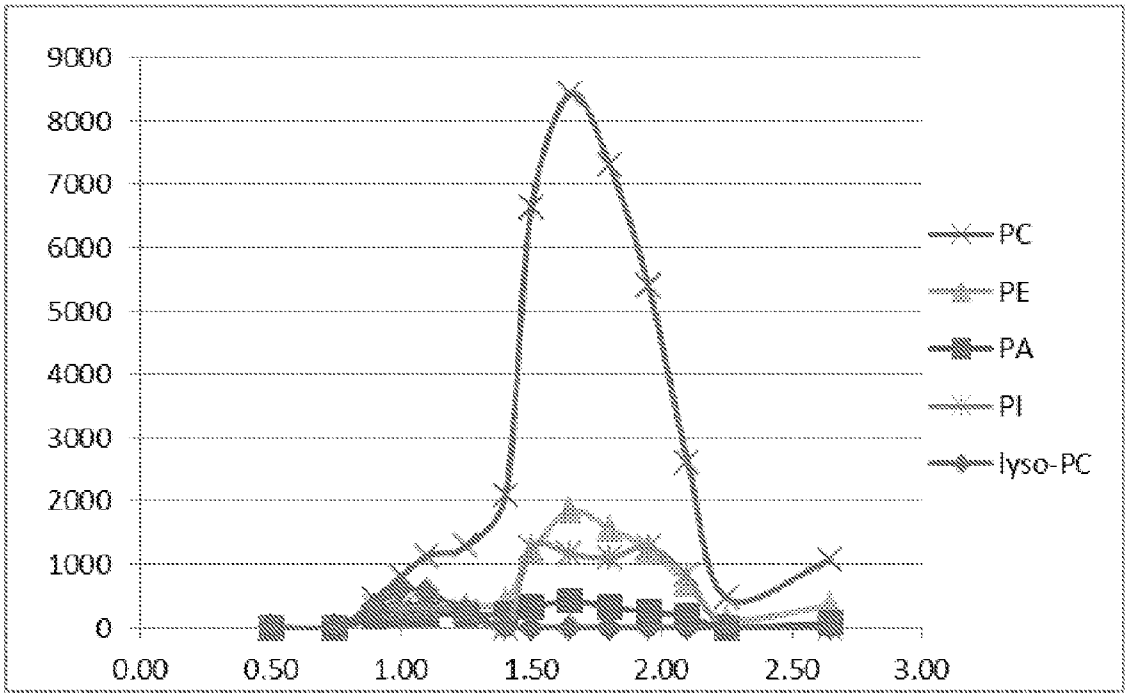
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
0.00
0.50
1.00
1.50
2.00
2.50
3.00
PC
PE
PA
PI
lyso-PC

FIGURE 13

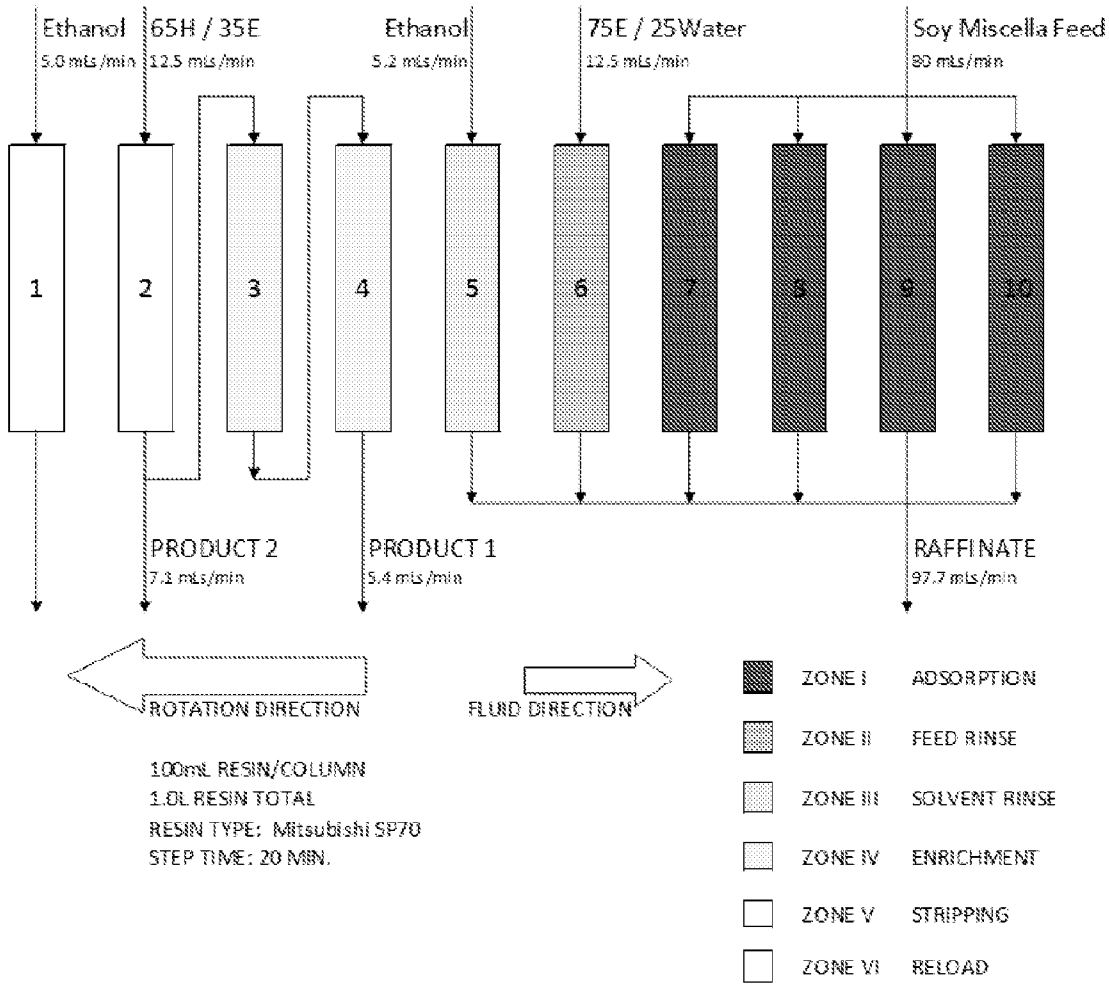
Ethanol
5.0 mLs/min
65H / 35E
12.5 mLs/min
Ethanol
5.2 mLs/min
75E / 25Water
12.5 mLs/min
Soy Miscella Feed
80 mLs/min
1
2
3
4
5
6
7
8
9
10
PRODUCT 2
7.1 mLs/min
PRODUCT 1
5.4 mLs/min
RAFFINATE
97.7 mLs/min
ROTATION DIRECTION
FLUID DIRECTION
100mL RESIN/COLUMN
1.0L RESIN TOTAL
RESIN TYPE: Mitsubishi SP70
STEP TIME: 20 MIN.
ZONE I ADSORPTION
ZONE II FEED RINSE
ZONE III SOLVENT RINSE
ZONE IV ENRICHMENT
ZONE V STRIPPING
ZONE VI RELOAD

FIGURE 14

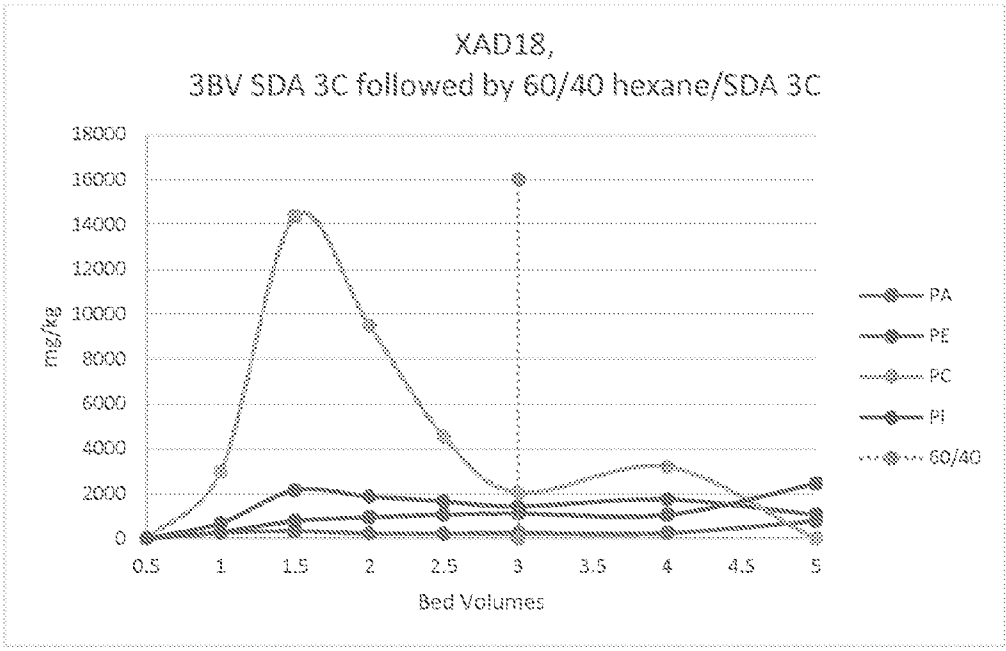
XAD18,
3BV SDA 3C followed by 60/40 hexane/SDA 3C
mg/kg
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
Bed Volumes
PA
PE
PC
PI
60/40

FIGURE 15

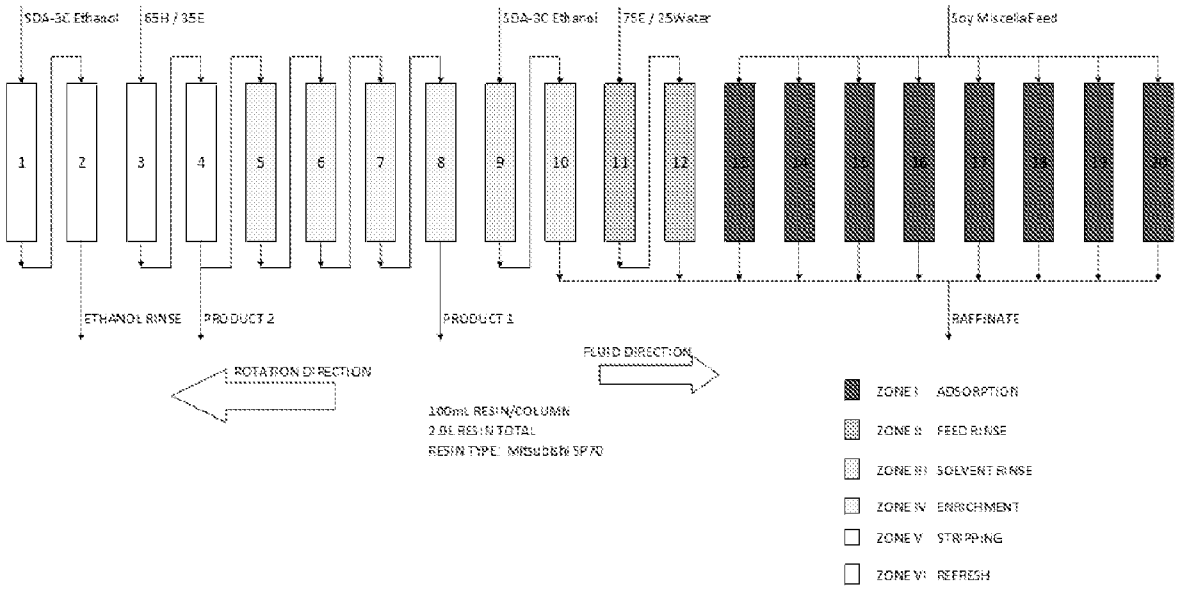
SDA-3C Ethanol
65H / 35E
SDA-3C Ethanol
75E / 25Water
Soy MiscellaFeed
1
2
3
4
5
6
7
8
9
10
11
12
ETHANOL RINSE
PRODUCT 2
PRODUCT 1
RAFFINATE
ROTATION DIRECTION
FLUID DIRECTION
100mL RESIN/COLUMN
2.8L RESIN TOTAL
RESIN TYPE: Mitsubishi SP70
ZONE I ADSORPTION
ZONE II FEED RINSE
ZONE III SOLVENT RINSE
ZONE IV ENRICHMENT
ZONE V STRIPPING
ZONE VI REFRESH

FIGURE 16

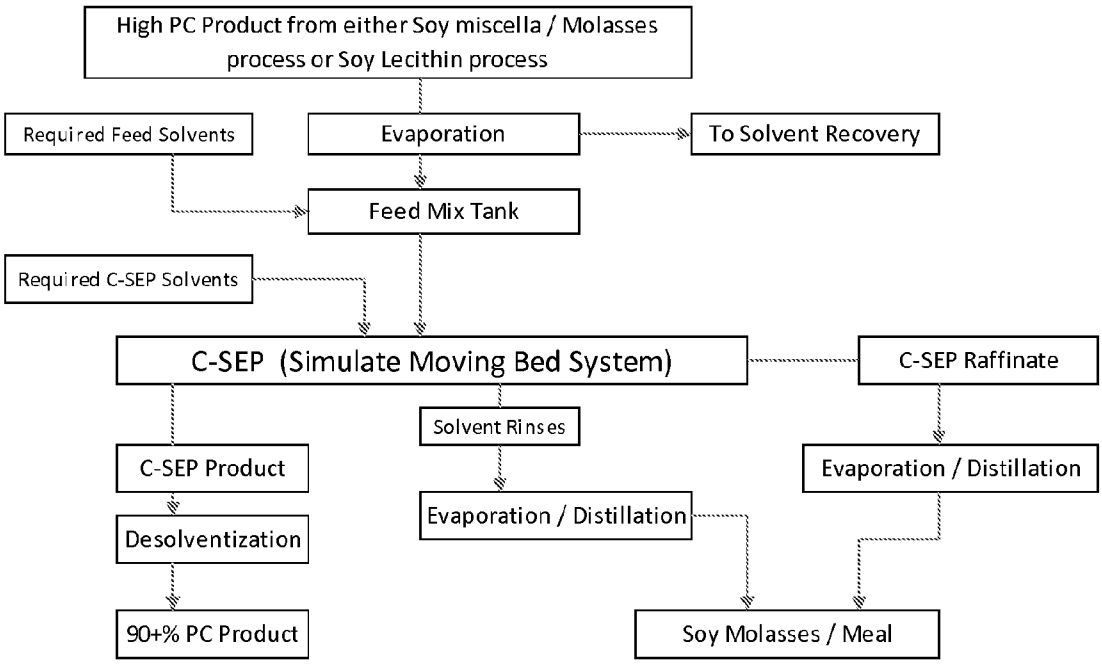
High PC Product from either Soy miscella / Molasses process or Soy Lecithin process
Required Feed Solvents
Evaporation
To Solvent Recovery
Feed Mix Tank
Required C-SEP Solvents
C-SEP (Simulate Moving Bed System)
C-SEP Raffinate
Solvent Rinses
C-SEP Product
Evaporation / Distillation
Evaporation / Distillation
Desolventization
90+% PC Product
Soy Molasses / Meal

FIGURE 17

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2015/064009

A. CLASSIFICATION OF SUBJECT MATTER
INV. A23J7/00 C07F9/10
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
A23J C07F

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	FR 2 388 823 A1 (NATTERMANN A & CIE [DE]) 24 November 1978 (1978-11-24) page 3, line 32 - page 5, line 23; examples 3,4	1-30
X	EP 0 115 981 A1 (SYNTHELABO [FR]) 15 August 1984 (1984-08-15) claims; example	1-30
X	EP 0 259 495 A1 (Q P CORP [JP]) 16 March 1988 (1988-03-16) claims; examples	1-30
X	US 2012/178950 A1 (SUGO KEN [JP]) 12 July 2012 (2012-07-12) claims	1-30

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 March 2016	21/03/2016

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

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2015/064009

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FR 2388823 A1	24-11-1978	AT 376880 B	10-01-1985
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		AU 3540978 A	01-11-1979
		BE 866461 A1	14-08-1978
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		DE 2718797 A1	02-11-1978
		DK 161478 A	28-10-1978
		ES 469227 A1	16-11-1978
		FR 2388823 A1	24-11-1978
		GB 1578109 A	29-10-1980
		IE 46626 B1	10-08-1983
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		NL 7804391 A	31-10-1978
		SE 431212 B	23-01-1984
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EP 0115981 A1	15-08-1984	AU 2391684 A	02-08-1984
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		GR 79755 B	31-10-1984
		IE 840234 L	01-08-1984
		JP S59152392 A	31-08-1984
		NO 840361 A	02-08-1984
		PT 78040 A	01-02-1984
		ZA 8400730 A	26-09-1984
EP 0259495 A1	16-03-1988	DK 532387 A	20-11-1987
		EP 0259495 A1	16-03-1988
		US 4847015 A	11-07-1989
US 2012178950 A1	12-07-2012	CN 102510896 A	20-06-2012
		DE 112010003779 T5	04-10-2012
		JP 5458239 B2	02-04-2014
		JP 2011068785 A	07-04-2011
		US 2012178950 A1	12-07-2012
		WO 2011037094 A1	31-03-2011