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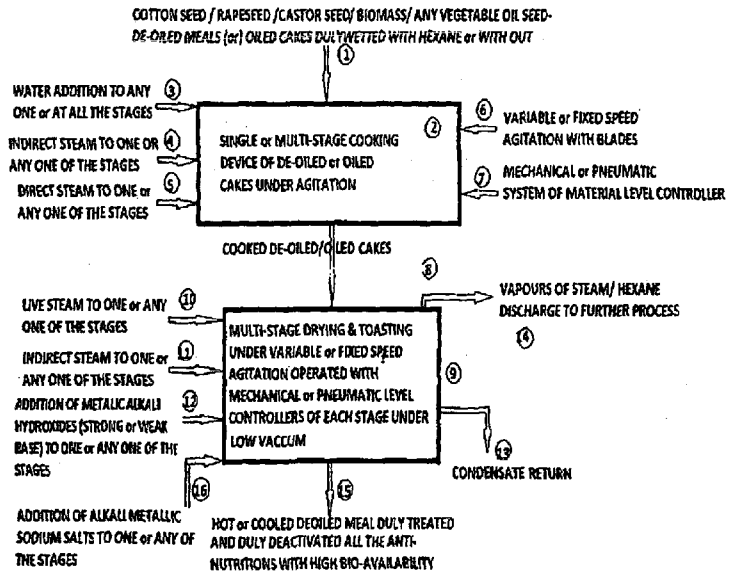
(54) Title: AN IMPROVED NOVEL PROCESS TO REDUCE ANTI-NUTRITIONAL AGENTS IN DE-OILED CAKE OF COTTON SEED MEAL/CASTOR SEED MEAL OR ANY OIL SEEDS MEAL/BIOMASS THEREOF

Process flow Chart of the Invented production of above,



(57) Abstract: a) The grinded de-oiled meal are taken into the single or multi-stage cooking device followed by agitation. b) To the processed meal obtained at step (a) a required quantity of water is been added at a single stage or at all the stages. c) After addition of water, an indirect steam treatment to be introduced in any one of the stage or at afl the stages and more number of treatments at different stages as depicted above. d) One can also have de-oiled meal after solvent extraction with hexane and perform the below steps to get the same result without performing steps a,b&c. e) Subjected de-oiled cake/meal of step a-c/d are treated in a DE-SOLVENTIZTION / TOASTER EQUIPMENT in presence of alkali hydroxides/ acides of strong or weak nature, in presence of direct heat and or steam, and thereby producing disintegrated, hydrolyzed, detoxified, de-solventized or dried and toasted Cottonseed/ Rapeseed/ Castorseed/ Rapeseen/ Castorseed de oiled cake or. Cottonseed/ Rapeseed/ Castorseed-oil cakes as the case may be. f) Drying the disintegrated hydrolyzed, detoxified, de-solventized or dried and toasted material obtained to step "e" to obtain dried, de-solventized and. toasted cake with improved properties. g) Alternatively, fortifying the dried, de-solventized and toasted cakes with one or more nutrients and energy rich compounds/additives either with or without adding of sodium salts of soapy slurry material and further. h) Application of said dried, de-solventized and toasted cakes with improved properties obtained in step "g" or fortified dried, de-solventized and toasted cake with one or more nutrients and energy rich compounds/additives as obtained in step T for animals, poultry, aqua...etc.

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Title: - An improved novel process to reduce anti - nutritional agents in de-oiled cake of cotton seed meal/ Castor seed meal or any oil seeds meal/Biomass thereof

Field of the invention: -

The present invention is to produce Cottonseed/ Rapeseed/ Castorseed/any kind of vegetable oilseed meal (oiled or de-oiled) or Biomass or in combination thereof to improve or modify the availability of valuable constituents and nutrients, detoxify by deactivating the anti-nutritional factors, and thereby improving their availability in a easily digestible or in dis-integrated form as an easy consumable and hence the applicability of said cake (oiled or de-oiled wetted with hexane or without wetted with hexane) for improving the health and productivity of animals, aqua etc.....and after application.

Back ground of the invention:-

This invention relates to a highly nutritious, easy digestible feed product/ingredients which is highly essential feeding product/ingredients required for feeding animals and poultry as the growing demands increases day by day for improving the quality and time bound weight development ,an essential requirement in the animal husbandry industry. Keeping in mind the present inventors have come with novel and inventive feed product/ingredients, which is easily digestible and high energetic as well as high nutritious compositions, which also provides instant energy for to the end users, as the invented process demonstrates and putting more efforts for breaking the high molecular

structure. The product also provides more bioavailability to the end users, and internal nourishing components of cotton seed meal, rape seed meal or castormeal or biomass or in combinations thereof having de-oiled cakes with or without solvent extraction of using normal hexane.

Objects of the invention: -

The object of the present invention which enhances the bio- availability of these ingredients effectively and gives full energy source of conversion and ultimately an all-round growth and health.

Further object of the present invention is that, it is essential to break down and/ or de-activate an anti-nutritional factors like Gossypol / Glucocinates / Resinins/ allergens components / and all its derivaties.

Further object of the present invention that the feed product can be effectively utilised by the end user without any wastage , but instantly releases of energy due to deactivation of anti-nutritional values of the de-oiled meal.

Description of the invention:-

The present invention pertains to improve or modify the availability of valuable constituents and nutrients, same time detoxify and deactivate the anti-nutritional factors, and thereby improving their availability in a easily digestible or in dis-integrated form as an easy consumable and hence the applicability of said cake/meal (oiled or de-oiled with hexane or without hexane) for improving the health and productivity of animals, aqua etc....,and after application. The said nutritious feed product comprising a solvent wetted de-oiled meal or direct de-oiled meal will be treated in the desolventization/ toasting equipment which is operating with an central agitator in a single or multiple staged, compartmentalised equipment while intake would be from its top & discharge at its bottom stage with any given rated capacity per hour, prior to this cooking of material would be done as well. The agitator speed could be fixed at constant or varying in order to maintain the variable or fixed residence time for the material from inlet to outlet.

The compartmentalized equipment had single or multiple number of stages where the material passes from top to down through a level controller device fitted either with mechanical or pneumatically system.

The cake material would go through an indirect steam heating through each compartment's double bottom arrangement at a varying pressure & flow rate as per the required quantity to heat the materials from 40 to 130 degree centigrade temperature

with a live steam arrangement at a variable flow and pressure rate at any given point of time depends upon the feed rate of material to be treated. Indirect or Direct live steam would be applied in all or few of the compartments based on the requirement. During the treatment process the de-oiled meal(with or without hexane) material would be

treated with alkali metallic hydroxides (week or strong base) at a feed rate of 1kgs to 50 kgs per each ton of cake material besides with or without the sodium salts of soapy slurry material to be added.

The aqueous solution fed and mixed with the cake would react and break the bonds of all the available amino acids, peptides, crude proteins, saccharides (mono, di, oligo, poly etc.), carbohydrates ,fatty acids, organic acids, vitamins, micro or macro nutrients and fibrous material into individual activative centers and the disintegrated components are be available to the animal at an easy form rather than using its energy & time to break in its gut which may not take place effectively as the above prevailing conditions are not exist.

The effective utilization of all these natural nutrients in oil cakes or de-oiled cake materials is determined by available form of various constituents and nutrients. When used as such, most of this material remains unutilized leading to generation of waste materials while consumed by the animal as these constituents are tightly bound the carbohydrate portion and therefore their availability after consumption requires longer retention times or is not completely available for effective growth and health of the animal.

So the invention displays the breakdown or disintegration of such de-oiled cake materials in the de-solvenisation / toasting device to produce useful and effective products yet in economic way is an essential and important task. Most of the processes

available in the literature requires slurring the Cottonseed cake/ de-oiled cake material, material for treatment and therefore consumes large amount of water, the treated Cottonseed cake / de-oiled cake mater cake materials or the Cottonseed/ Rapeseed/Castorseed-/ becomes difficult to filter and dry and hence becomes economically and commercially unviable. Further, the crude Cottonseed cake/ de-oiled

cake or any of this material contains numerous toxin substances for example Gossypol/Glucocinates/Resinins/Cylcopropanoid fatty acids, pesticides...etc, and hence cannot be used directly for particular consumption as food or feed to the animals. Also the yields of products produced by such processes are low and products are not'in useful form for said application.

Therefore the aim of the present invention is to device a multi-objective process for treatment of said Cottonseed cake/ Rapeseed cake/ Castorseed cake / de-oiled meal (with or without hexane) to improve or modify the availability of valuable constituents and nutrients, detoxify and deactivate the anti-nutritional factors, and thereby improving their availability in a easily digestible or in dis-integrated form as an easy consumable and hence the applicability of said cake/meal (oiled or de-oiled with hexane or without hexane) for improving the health and productivity of animals, aqua etc..... and after application.

In an embodiment of the process of present invention the Cottonseed/ Rapeseed/ Castprseed-cake/meal or other material is obtained from Cottonseed/ Rapeseed/ Castorseed-/ de-oiled cake or oil cake an agricultural sources and is natural or modified (such as but not limited to hybrids, transgenic, genetically modified).

In another embodiment of the process of the invention said Cotton seed/ Rapeseed/ Castorseed- cake is subjected to one or more treatments selected from a group but not limited to micronization, pulverization, grinding, crushing , dehulling, flaking, expanding, extrusion, milling, expelling/extraction or suitable combinations thereof: with or without heating and / or wetting; in presence or absence of aqueous, organic, aqueous-organic solvent/s optionally containing an additive/s and / or co-solvent/s.

In order to achieve the said process the present inventors have developed an unique method of making nutritional feed product comprising the following steps:-

a) The grinded de-oiled meal are taken into the single or multi-stage cooking device followed by agitation.

b) To the processed meal obtained at step (a) a required quantity of water is been added at a single stage or at all the stages.

c) After addition of water, an indirect steam treatment to be introduced in any one of the stage or at all the stages and more number of treatments at different stages as depicted above.

d) One can also have de-oiled meal after solvent extraction with hexane and perform the below steps to get the same result without performing steps a,b&c.

e) Subjected de-oiled cake/meal of step a-c/d are treated in a DE-SOLVENTIZTION / TOASTER EQUIPMENT in presence of alkali hydroxides/ acides of strong or weak nature, in presence of direct heat and or steam, and thereby producing disintegrated, hydrolyzed, detoxified , de-solventized or dried and toasted Cottonseed/ Rapeseed/ Castorseed/ Rapeseen/ Castorseed de oiled cake or Cottonseed/ Rapeseed/ Castorseed-oil cakes as the case may be.

f) Drying the disintegrated hydrolyzed, detoxified, de-solventized or dried and toasted material obtained to step "e" to obtain dried, de-solventized and toasted cake with improved properties.

g) Alternatively, fortifying the dried , de-solventized and toasted cakes with one or more nutrients and energy rich compounds/additives either with or without adding of sodium salts of soapy slurry material and further.

h) Application of said dried, de-solventized and toasted cakes with improved properties obtained in step "g" or fortified dried, de-solventized and toasted cake with one or more nutrients and energy rich compounds/additives as obtained in step T for animals, poultry, aqua...etc.

Example

The present invention pertains to treatment with alkali (weak or stong base) to improve or modify the availability of valuable constituents and nutrients, detoxify and deactivate the anti-nutritional factors, and thereby improving their availability in a easily digestible or in dis-integrated form as an easy consumable and hence the applicability of said cake/meal (oiled or de-oiled with hexane or without hexane) for improving the health and productivity of animals, aqua etc.....and after application there of which the process established in large commercial scale ;

The adding of alkali (weak or strong base) shall have a different concentration varying from 1 to 60% (%w/w),the reaction of hydrolysis takes place while during the toasting / drying temperatures duly maintained at 80 °C to 100 °C in particular and could be varying from 40 °C to 130 °C. Also it is to be noted that the feed rate of quantity of metallic alkali hydroxides would be added at 1 kgs / ton to 50 kg/ton of de-oiled cakes / bio mass with or without addition of soap stock material produced during neutralization of edible oils, same time .

The spraying or admixing of metallic alkali hydroxides of varying concentration into the above de oiled cakes / oil cakes / bio mass (of cottonseed, rapeseed, castorseed or any bio mass materials) in presence of live and indirect steam where the hydrolysis took place at a constant agitation and releases the free amino acids and peptides to maximum extent. In addition to that the anti nutritional elements gets neutralized or deactivated (aflatoxins / gossypols in case of cottonseed, glucosinolates in case

rapeseed , resins in case of castor respectively) and improvisation the digestibility and bio availability to the animal or birds. The process further also reduces the heat requirement as due to the exothermic reaction tookplace and evaporates any kind of solvents if are exist and economizes the process to a large extent:

We Claim,

1. An improved process to reduce anti nutritional agents in de-oiled meal of cottonseed meal or Rapeseed meal or Castorseed meal or any biomass, with improved availability of valuable constituents and nutrients comprising of the following steps:

a) The de-oiled meal (grinded or ungrinded) are taken into the single or multi-stage cooking device followed by agitation.

b) To the processed meal obtained at step (a) a required quantity of water has been added at a single stage or at all the stages.

c) After addition of water, an indirect steam treatment to be introduced in any one of the stage or at all the stages and more number of treatments at different stages as depicted, above.

d) One can also have de-oiled meal after solvent extraction with hexane and perform the below steps to get the same result without performing steps a,b&c.

e) Subjected de-oiled cake/meal of step a-c/d are treated in a DE-SOLVENTIZTION / TOASTER EQUIPMENT in presence of alkali of strong or weak nature, in presence of direct heat and or steam, and thereby producing disintegrated, hydrolyzed, detoxified , de-solventized or dried and toasted Cottonseed/ Rapeseed/ Castorseed/ de oiled /meal.

f) Drying the disintegrated hydrolyzed, detoxified, de-solventized or dried and toasted material obtained to step "e" to obtain dried, de-solventized and toasted cake with improved properties.

g) Alternatively, fortifying the dried, de-solventized and toasted cakes with one or more nutrients and energy rich compounds/additives either with or without adding of sodium salts of soapy slurry material and further.

h) Application of said dried, de-solventized and toasted cakes with improved properties obtained in step "g" or fortified dried, de-solventized and toasted cake with one or more nutrients and energy rich compounds/additives as obtained in step "f for animals, poultry, aqua...etc.

2. A method as claimed in claim 1, wherein the Desolventization / Toasting & Hydrolysis of Proteins by adding of Metalic Alkaii Hydroxides of (weak or strong base) to
the oil extracted hexane wetted meal or meal based materials, to get effective complex
proteins structure duly broken down, with high level of availability of peptides, free amino acids.

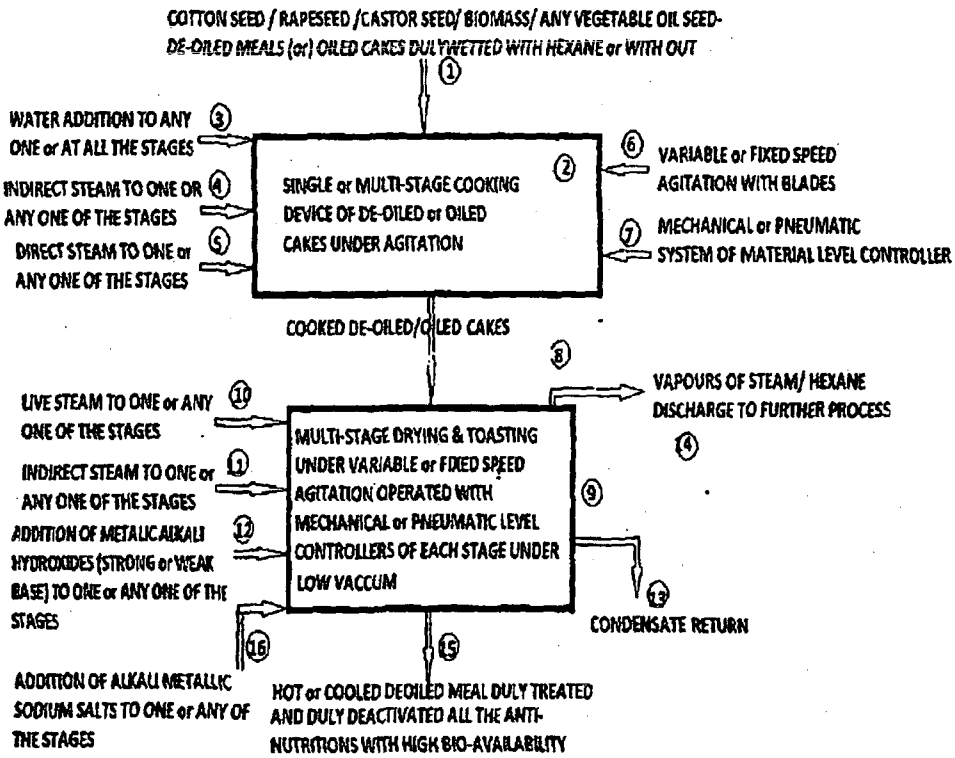
3. A method as claimed in claim 1, wherein the Desolventization/Toasting & Hydrolysis of carbohydrates and fibres ingredients, by adding of Metalic Alkali Hydroxides of (weak or strong base) to the oil extracted hexane wetted meal or meal
based materials, to get effective complex carbohydrate and fibrous structure duly broken down, with high level of availability of simple carbohydrates and fibre..

4. A method as claimed in claim 1 and 2, wherein the metallic alkali hydroxides (weak or strong based) shall have a different concentration varying from 1 to 60% (%w/w),the reaction of hydrolysis takes place while during the toasting / drying temperatures duly maintained at 80 °C to 130 °C in particular from 40 °C to 100 °C.

5. A method as claimed in claim 3, wherein the quantity of metallic alkali hydroxides added at 1 kgs / ton to 50 kg/ton with or without addition of soap stock material

6. A method as claimed in claim 2/3/4, wherein the metallic alkali hydroxides is added in presence of live and indirect steam at a constant agitation, to deactivate anti nutritional agents

Process flow Chart of the Invented production of above,



INTERNATIONAL SEARCH REPORT

International application No
PCT/IN2014/000513

A. CLASSIFICATION OF SUBJECT MATTER
INV. A23K1/00 A23K1/14 A23K1/16 A23K1/18 A23L1/211
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)
A23K A23L A61K

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, FSTA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1 575 506 A (UNILEVER LTD) 24 September 1980 (1980-09-24) claims 1-11; example 1	1-6
A	EP 0 006 654 A2 (UNILEVER NV [NL]; UNILEVER PLC [GB]) 9 January 1980 (1980-01-09) the whole document	1-6
X	US 2 958 600 A (THURMAN BENJAMIN H) 1 November 1960 (1960-11-01) column 5, lines 18-56; claims 1-16	1-6
A	US 2 934 431 A (CAVANAGH GEORGE C) 26 April 1960 (1960-04-26) the whole document	1-6

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

☒ See patent family annex.

* Special categories of cited documents :

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

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"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

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
9 January 2015	21/01/2015

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IN2014/000513

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 1575506 A	24-09-1980	BE 850943 A1	01-08-1977
		CA 1091085 A1	09-12-1980
		DE 2704743 A1	11-08-1977
		DK 43677 A	07-08-1977
		FI 770310 A	07-08-1977
		FR 2340059 A1	02-09-1977
		GB 1575506 A	24-09-1980
		IE 44519 B1	30-12-1981
		IT 1072899 B	13-04-1985
		LU 76708 A1	18-08-1977
		NL 7701124 A	09-08-1977
		NO 770385 A	09-08-1977
		PL 195819 A1	02-01-1978
		SE 424691 B	09-08-1982
EP 0006654 A2	09-01-1980	CA 1102172 A1	02-06-1981
		DE 2962386 D1	06-05-1982
		DK 267179 A	27-12-1979
		EP 0006654 A2	09-01-1980
		FI 791962 A	27-12-1979
		US 4244973 A	13-01-1981
US 2958600 A	01-11-1960	NONE	
US 2934431 A	26-04-1960	NONE	