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(54) Title: A PROCESS FOR THE RECOVERY OF DE-SALTED ORGANICS FROM SPENT WASH SUITABLE FOR UTILIZATION IN CATTLE FEED FORMULATION

(57) Abstract: Sugarcane molasses based alcohol distilleries generate dark coloured effluent commonly known as spent wash. Due to its high potassium content; ca. 10% on dry matter basis, spent wash is not preferred for use in cattle feed formulation. The present invention provides a process for the recovery of low potassium containing organics ($K^+ < 3\%$ on dry matter basis) from spent wash and methods for its utilisation, as binder in cattle feed formulation, in place of sugarcane molasses. Then instant invention would enable value addition and utilisation of sugarcane molasses based distillery effluent and in effect would make additional quantity of sugarcane molasses, erstwhile consumed in preparation of cattle feed, available for alcohol production.



A PROCESS FOR THE RECOVERY OF DE-SALTED ORGANICS FROM SPENT WASH SUITABLE FOR UTILIZATION IN CATTLE FEED FORMULATION

FIELD OF THE INVENTION

5 The present invention relates to a process for the recovery of de-salted organics from spent wash suitable for utilization in cattle feed formulation. In particular, the present invention provides a process for the recovery of organics containing less than 3% potassium on dry matter basis from the effluent generated in sugarcane molasses based alcohol distillery effluent; commonly known as spent wash/ vinasse and methods for its utilization in cattle feed
10 formulation as binder. In a broader perspective this invention enables value recovery from spent wash (SW) a waste material.

BACKGROUND OF THE INVENTION AND DESCRIPTION OF PRIOR ART

Indian sugar mills produce about 12-14 million tonnes [t] molasses annually. Distilleries
15 convert about 9-10 million t of this molasses to generate 2.5 million m³ alcohol. Significant quantity of molasses is also utilized in cattle feed formulation as binder. However, with increasing thrust in Government of India's Ethanol Blending Programme, molasses availability for non-ethanol/alcohol applications, viz., cattle feed etc. is likely to be reduced significantly. In such scenario, there is a need to develop alternate binder to replace molasses for cattle feed
20 formulation.

Although concentrated (50-60% TS) spent wash (distillery effluent) exhibits reasonable binding properties, its high potassium content (around 10% w/w on dry matter basis) is known to adversely affect health & productivity of the cattle & necessitates corrective measures.

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Potassium is an essential component for dairy cows. While diet without adequate potassium results in almost immediate drop in milk production, high concentration of potassium results in:

- 1) reduced absorption of magnesium
- 30 2) increased phosphorus deficiency
- 3) low concentration of calcium in blood, leading to hypocalcemia milk fever, udder edema etc.

Hence it is important to monitor & regulate potassium level in cattle feed.

Sugarcane molasses based alcohol distillery effluent is an extremely complex system, containing various compounds (viz., melanoidins, caramels, traces of sugar etc.) and ions (viz., potassium, calcium, sulphate, chloride etc.). Over the years, several efforts have been made to harness nutritive values (viz., crude proteins, crude fibers, crude fats, minerals etc.) of SW by incorporating it in cattle feed. However, high potassium content of SW (ca. 10% on dry matter basis) put severe limitation on utilisation in cattle feed formulation.

Reference may be made to the research article “Using strong acid–cation exchange resin to reduce potassium level in molasses vinasses” by M. Decloux et. al. ([https://doi.org/10.1016/S0011-9164\(02\)00520-9](https://doi.org/10.1016/S0011-9164(02)00520-9)) which teaches about method for partial removal of potassium from beet molasses based distillery effluent through electrodialysis technique. Although experimental results indicate significant reduction of potassium concentration, various operational issues viz., membrane fouling, reduced flux etc. are likely to pose serious limitation in large scale implementation of the process.

Reference may be made to the research article “Interest of electrodialysis to reduce potassium level in vinasses - Preliminary experiments” by P.-J. Zhang et al (<https://doi.org/10.1016/j.desal.2011.11.024>) which teaches about method for removal of potassium from sugarcane molasses based distillery effluent through cation exchange technique. Resin fouling on account of high organic loading of effluent, high energy requirement for evaporation of dilute potash containing elute are some of the constraints in large scale implementation of the process.

Reference may be made to the patent application “method for reducing potassium content in vinasse, its use, precipitate and process solution” bearing international publication number WO2013087969 A1 dated 20th June 2013, by Vuori A., which teaches about method for partial removal of potassium from vinasse through precipitation of potassium sulphate. Extent of potassium removal, achieved upon implementation of this method, is about 30 – 40%, even while using vinasse with relatively high K⁺ concentration (ca. 3.5%). Further, incorporation of ammonia / ammonium ion in liquid stream is likely to render the potassium depleted aqueous stream unfit for utilisation in cattle feed formulation.

While above mentioned methods operate around the pivotal theme of reduction of potassium content is vinasse, another approach is to separate & recover the organics from spent wash stream, leaving K^+ in aqueous phase.

- 5 Reference may be made to patent application no. PCT/IN2018/050100 dated 26th February, 2018 by Maiti P. et al. which teaches about a method for partial removal of organics from SW as carbon-rich sludge. Although relatively free from potassium (ca. <1% on dry matter basis), this sludge is highly alkaline due to presence of large quantity of free lime & is not suitable for utilization in cattle feed formulation as such.

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Thus, keeping in view the drawbacks of the hitherto reported prior art, the inventors of the present invention realized that there exists a dire need to devise a method for the recovery of desalted organics (DSO) from spent wash, suitable for utilization in cattle feed formulation to entirely substitute molasses as binder in cattle feed formulations.

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OBJECTIVES OF THE INVENTION

The main object of the present invention is therefore to provide a method for the utilization of the organics present in spent wash, in lieu of molasses, in cattle feed formulation as a binder.

- 20 Another object of the present invention is to devise a process for recovery of desalted organics from spent wash which are suitable for utilisation in cattle feed formulation.

Still another object of the present invention is to utilize the desalted organics in cattle feed formulation in place of molasses while retaining mechanical strength of the feed pellets.

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Yet another object of the present invention is to derive economic value from spent wash through generation of value-added by-products, viz., desalted organics.

SUMMARY OF THE INVENTION

- 30 The present invention relates to a process for obtaining desalted organics from spent wash which are useful as a binder for Cattle/Poultry Feed formulation as an alternative to molasses. Efficacy, palatability and plant scale trial studies of the developed cattle feed formulation was done at major dairy research institutes, namely, National Dairy Research Institute, Karnal

(HR); National Dairy Development Board, Anand (GJ); Sabar Dairy, Himmatnagar (GJ). The results indicated that the cattle feed formulation containing desalted organics in place of molasses exhibited mechanical strength of the feed pellets comparable to those obtained using molasses.

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In an embodiment, the present invention provides a process for the recovery of desalted organics from spent wash, suitable for utilisation in cattle feed formulation as complete replacement of sugarcane molasses. The process comprises the major steps of lime treatment of spent wash to generate carbon-rich sludge following the general procedures as disclosed in prior art; mixing the carbon-rich sludge with water, under stirring, to prepare slurry; addition of carbon dioxide gas into the slurry to reduce the pH to around 8.5; separation of organic rich liquid and precipitated calcium carbonate; addition of sulphuric acid into the organic rich liquid till pH is around 4.0; separation of organic rich liquid and precipitated gypsum; evaporation of the organic rich liquid till solid concentration reaches 50-60% to obtain desalted organics suitable for cattle feed formulation; calcination of the precipitated calcium carbonate to produce calcium oxide & carbon dioxide; recycling of the calcium oxide; and recycling of the carbon dioxide.

In another embodiment, the present invention provides a process for the recovery of desalted organics from spent wash, suitable for utilisation in cattle feed formulation as complete replacement of sugarcane molasses, wherein the said process comprising the steps of:

- (i) flocculation of colloidal organic particulates by addition of lime (calcium oxide / hydroxide) into SW (pH: 4.2), under stirring, till pH is ca. 12-13;
- (ii) separation of flocculated organics, from the aqueous phase, as carbon rich sludge obtained upon filtration of the slurry of step (i);
- (iii) addition of the carbon rich sludge from step (ii) into water, under stirring, to prepare slurry;
- (iv) converting free lime, present in flocculated organics, into calcium carbonate by addition of carbon dioxide gas into the slurry of step (iii) to reduce pH to ca. 8.5-9;
- (v) filtration of the slurry of step (iv) to separate organic rich liquid and solid calcium carbonate;
- (vi) removal of Ca^{2+} ion present in solution, as insoluble gypsum, by addition of sulphuric acid into the organic rich liquid from step (v) till pH is ca. 4-5;

- (vii) separation of organic rich liquid and precipitated gypsum;
- (viii) evaporation of the organic rich liquid from step (vii) till solid concentration reaches 50-60% to obtain desalted organics suitable for cattle feed formulation;
- (ix) calcination of the precipitated calcium carbonate from step (v) to produce calcium oxide & carbon dioxide;
- (x) recycling of the calcium oxide from step (ix) to step (i);
- (xi) recycling of the carbon dioxide from step (ix) to step (iv).

In still another embodiment, the present invention provides a process, wherein lime used is selected from calcium oxide or calcium hydroxide.

In yet another embodiment, the present invention provides a process, wherein the acid used is selected from sulphuric acid or phosphoric acid.

In still another embodiment, the present invention provides a process, wherein potassium content in de-salted organics is less than 3% on dry matter basis.

In still another embodiment, the present invention provides a process, wherein raw material consumption is minimized through calcination of precipitated calcium carbonate and recycling the resultant calcium oxide & carbon dioxide.

In still another embodiment, the present invention provides a process, wherein the desalted organics obtained in step [g] are useful in the preparation of cattle feed formulation.

In another embodiment, mechanical strength & durability of cattle feed pellets formulated using desalted organics was found to be comparable to the pellets prepared using of sugarcane molasses.

In another embodiment, de-mineralization of carbon rich sludge was effected by using phosphoric acid, in place of sulphuric acid.

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS

Figure-1 represents the overall scheme of the claimed process for recovery of desalted organics (DSO) from spent wash (SW), suitable for utilisation in cattle feed formulation, in lieu of molasses.

DETAILED DESCRIPTION OF THE INVENTION

Sugarcane molasses based alcohol distilleries generate dark coloured effluent, commonly known as spent wash. Due to its high potassium content; around 10% on dry matter basis, the

spent wash is not preferred to be used in cattle feed formulation. The present invention provides a process for the recovery of low potassium containing organics ($K^+ < 3\%$ on dry matter basis) from spent wash and methods for its utilization, preferably as binder in cattle feed formulation, in place of sugarcane molasses. The present invention would enable value addition and utilization of sugarcane molasses based distillery effluent and in effect would make additional quantity of sugarcane molasses, erstwhile consumed in preparation of cattle feed, available for alcohol production.

The present invention provides a process for the recovery of desalted organics (DSO) from sugarcane molasses based alcohol distillery effluent (spent wash, SW) useful in cattle feed formulation as binder wherein the said process comprising the steps of:

- [a] adding lime into spent wash having pH 4.2 under stirring, till pH is ca.12.5;
- [b] separating carbon rich sludge as solid residue after filtration of the slurry obtained in step [a];
- [c] adding the carbon rich sludge of step [b] in water under stirring to prepare a slurry;
- [d] adding carbon dioxide gas into the slurry of step [c] to reduce the pH to ca. 8.5;
- [e] filtering the slurry obtained in step [d] to separate organic rich liquid-1 and solid calcium carbonate;
- [f] adding acid to the organic rich liquid-1 as obtained in step [e] till pH is ca. 4 and separating the organic rich liquid-2 while precipitating gypsum;
- [g] evaporating the organic rich liquid-2 obtained in step [f] till the solid concentration reaches 50-60% to obtain desalted organics suitable for cattle feed formulation;
- [h] calcining the precipitated calcium carbonate from step [e] to produce calcium oxide & carbon dioxide;
- [i] recycling the calcium oxide obtained in step [h] in step [i];
- [j] recycling the carbon dioxide from step [h] in step [d].

INVENTIVE STEPS OF THE PRESENT INVENTION

- (i) The main inventive step is the development of a process for recovery of desalted organics from spent wash, suitable for utilization in cattle feed formulation as binder.
- (ii) Another inventive step is demineralization of potassium free carbon rich sludge ($K < 1\%$ on dry matter basis), obtained from spent wash following the general procedures as

disclosed in prior art, through precipitation and separation of calcium carbonate and gypsum.

(iii) Another inventive step is to completely substitute sugarcane molasses in cattle feed formulation by the desalted organics, and retaining mechanical strength of the feed pellets.

(iv) Another inventive step is to reduce raw material consumption in the process by utilising the calcium oxide, obtained upon calcination of the precipitated calcium carbonate, for generation of carbon rich sludge from spent wash.

10 EXAMPLES

The following examples are given by way of illustration only and therefore should not be construed to limit the scope of the present invention in any manner.

Example-1

15 1 L spent wash [dark brown, opaque, K^+ :1.01 % (w/v)] was reacted with 50 gm calcium hydroxide [available lime: 80% (w/w)] under stirring. pH of the reaction mixture was 13.1. Stirring was continued for 3 hrs. Upon centrifugation of the resultant slurry, 200 gm wet cake and 0.85 L filtrate [reddish brown, transparent, pH: 12.9, Ca^{2+} : 1.2%, K^+ : 1.01% (w/v)] was obtained. The wet cake was air dried to obtain 150 gm organic rich solid (Ca^{2+} : 10.7%, K^+ :
20 0.68% w/w).

Example-2

100 gm organic rich solid, obtained following the procedure of example-1, was mixed in 200 mL water under stirring. Carbon dioxide gas was purged into the resultant slurry to lower &
25 maintain pH of the reaction mixture to 8.6. Stirring was continued for 2 hours. Upon centrifugation of the reaction mixture, 110 gm wet cake (calcium carbonate, Ca^{2+} : 15.3% w/w) and 150 mL filtrate [reddish brown, transparent, pH: 8.5, Ca^{2+} : 0.84%, K^+ : 0.58% (w/v)] was obtained.

Example 2 teaches us the method of separation of free lime from organic rich liquid through
30 precipitation of calcium carbonate.

Example-3

Previously prepared dilute solution of sulphuric acid [50% (w/v)] was slowly added into 100 mL filtrate [reddish brown, transparent, pH: 8.5, Ca^{2+} : 0.8%, K^+ : 0.6% (w/v)], obtained following the procedure of example-2, under stirring to lower pH of reaction mixture to 4.0.

5 Stirring was continued for 2 hours. Upon centrifugation of the reaction mixture, 7 gm wet cake [gypsum, Ca^{2+} :8.6% (w/v)] and 95 mL filtrate [reddish brown, transparent, pH: 4.0, Ca^{2+} : 0.2%, K^+ : 0.6% (w/v)] was obtained.

Example 3 teaches us the method to remove soluble calcium ion from organic rich liquid through precipitation of gypsum.

Example-4

Previously ground & screened ingredients (size 0.5-0.7 mm) comprising of wheat grain (20 kg), maize grain (23 kg), rice grain (18 kg), groundnut cake (10 kg), mustard cake (20 kg) & salt (sodium chloride, 1 kg) were mixed thoroughly & conditioned by injecting live steam to the mixture. Subsequently warm ($50 \pm 5^\circ\text{C}$) sugarcane molasses (containing 60% total solids, 8 kg) was added to the mixture & mixing was continued for another 30 minutes, to achieve uniform consistency. The whole mass was then subjected to extrusion, maintaining the temperature at $70 \pm 5^\circ\text{C}$. Extruded pellets were further cooled to ambient temperature using forced draft air circulation, prior to bagging.

Example-5

Feed pellets were prepared following the formulation & procedure of example-4, except utilisation of desalted organics [filtrate from example-3 concentrated through evaporation to 60% total solids, Ca^{2+} : 0.48%, K^+ : 1.62% (w/v)] in place of sugarcane molasses.

Pellet Durability Index (PDI) of the pellets of both example 4 & 5 were evaluated using Holmen tester. While PDI of sugarcane molasses based pellets (from example-4) was 95.715, that of desalted organics based pellets (from example-5) was 97.035.

Example 4 & 5 demonstrate that desalted organics can be used to substitute sugarcane molasses in cattle feed formulation without affecting mechanical strength of the feed pellets.

Example-6

Previously prepared dilute solution of phosphoric acid [50% (w/v)] was slowly added into 100 mL filtrate [reddish brown, transparent, pH: 8.5, Ca^{2+} :0.8%, K^+ :0.5% (w/v)], obtained following the procedure of example-2, under stirring, to lower pH of reaction mixture to 5.0.

- 5 Stirring was continued for 2 hours. Upon centrifugation of the reaction mixture, 6 gm wet cake [calcium phosphate, Ca^{2+} : 10.2% (w/v)] and 95 mL filtrate [reddish brown, transparent, pH: 5.0, Ca^{2+} : 0.2%, K^+ : 0.5% (w/v)] was obtained.

Example 6 teaches us that removal of soluble calcium ions can also be effected with phosphoric acid, in lieu of sulphuric acid, through precipitation of calcium phosphate.

10 **Example 7**

Cattle feed pellets produced in Nand Gopala Feeds - a commercial plant at Sagbara, Gujarat – 393 050.

Ingredients: Maize, Wheat Grain, Groundnut Cake, Rapeseed Meal, Wheat Bran.

Binder: De-potash Vinasse and Molasses

- 15 The CF Feed Pellets with DPV and molasses (4.5 ton of each batch) were utilised for animal study.

The study was conducted to investigate the effect of De-Potash Vinasse (DPV) as pellet binder on nutrient utilization, blood biochemical parameters and production performance in Murrah
20 buffaloes. The study conducted /carried out in two phases i.e., *in vitro* and *in vivo* experiment.

In vitro experiment was conducted to observed the effect DPVs on rumen fermentation kinetics viz., dry matter digestibility (IVDMD), organic matter digestibility (IVOMD), ammonia nitrogen ($\text{NH}_3\text{-N}$), partition factor (PF) and microbial biomass production (MBP). For
25 comparison, concentrate mixture, pellet with molasses (Pellet M) and pellet with depotash vinasse (Pellet V) was prepared with same ingredients. The binders viz., molasses and DPV was added @8%. The substrate for the *in vitro* experiment comprised of concentrate, Pellet M, Pellet V and total mixed rations (TMRs) with concentrate/ pellets, oat green, wheat straw in different proportions (40:40:20). The results revealed no noticeable changes occurred due to
30 addition of DPV pellets on rumen fermentation parameters. *In vitro* DM, OM and CP digestibility was similar in all the groups. Microbial biomass production and ammonia nitrogen was also similar in all the groups. This indicated that DPV had no negative impact on rumen fermentation, microbial population and nutrient utilization.

In vivo trial with fifteen early lactating Murrah buffaloes were randomly distributed on the basis of milk yield and days in milk in three groups *viz.*, control, T₁ and T₂. Animals in control group fed basal diet consisting of concentrate mix, oat green and wheat straw; animals T₁ groups supplemented with Pellet M with oat green and wheat straw while T₂ animals were fed Pellet V with oat green and wheat straw.

Blood samples were collected at the onset of experimental feeding and thereafter at 60 day and 120 days interval for biochemical parameters and minerals. A metabolic trial of seven days collection period was conducted on all the buffaloes to study the digestibility and balance of nutrients at the end of the experiment. Dry matter intake (DMI) and milk yield recorded daily and its composition determined fortnightly basis.

During the experimental trial, there was no change in body weight and body condition score due to dietary treatment. DMI (kg/d and % of BW) was found to be similar ($P>0.05$) among the groups. There was no effect of dietary treatment on digestibility of any of the nutrients and total digestible nutrients (TDN) were similar. Nitrogen balance as percent of intake and absorbed nitrogen did not vary significantly ($P>0.5$) among the control and treatment groups. The plasma glucose, total protein, albumin, blood urea nitrogen (BUN), total cholesterol and hepatic enzymes *viz.*, alanine amino transferase (ALT) and aspartate amino transferase (AST) level were within normal biological range and statistically ($P>0.05$) similar.

The mean value of plasma Ca, Mg, Na, K, Zn, Cu and Fe were not altered with molasses and DPV pellets supplementation. There were no changes observed in milk yield and its composition. The feeding of pellet with DPVs as pellet binder@8% to the early lactating buffaloes didn't have any adverse effect on blood biochemical parameters and plasma minerals, milk yield and its composition.

ADVANTAGES OF THE PRESENT INVENTION

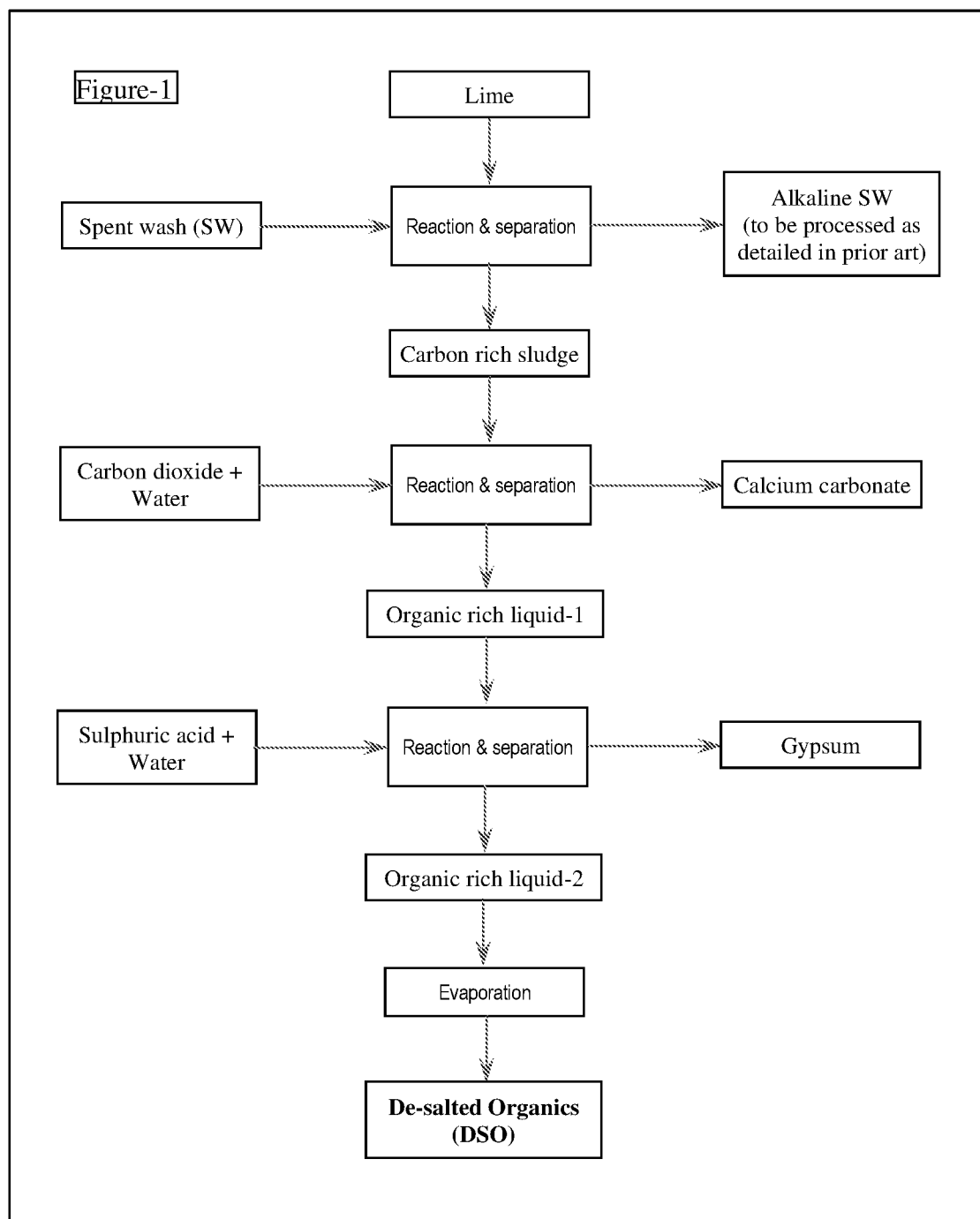
- The present invention provides a process for recovery of desalted organics from spent wash, suitable for utilisation in cattle feed formulation as complete replacement of sugarcane molasses, thereby enabling realization of additional economic benefit through recovery of value-added by product from alcohol distillery effluent, and recovery of

additional quantity of ethanol, an increasingly important renewable fuel, from the sugarcane molasses erstwhile utilized in cattle feed formulation.

- Since the desalted organics obtained is relatively free from potassium & other inorganic constituents, it does not pose dosage limitation in cattle feed formulation vis-à-vis spent wash.
- Desalted organics based feed pellets exhibit comparable mechanical strength vis-à-vis molasses based pellets.
- Except the final concentration step, the core process of spent wash treatment and subsequent recovery of desalted organics, as disclosed in the present invention, operate at ambient temperature (20–35 °C) and does not pose major operational difficulties.

WE CLAIM

1. A process for the recovery of desalted organics (DSO) from sugarcane molasses based alcohol distillery effluent (spent wash, SW) useful in cattle feed formulation as binder wherein the said process comprising the steps of:
 - 5 [a] adding lime into spent wash having pH 4.2 under stirring , till pH is ca.12.5;
 - [b] separating carbon rich sludge as solid residue after filtration of the slurry obtained in step [a];
 - [c] adding the carbon rich sludge of step [b] in water under stirring to prepare a slurry;
 - [d] adding carbon dioxide gas into the slurry of step [c] to reduce the pH to ca. 8.5;
 - 10 [e] filtering the slurry obtained in step [d] to separate organic rich liquid-1 and solid calcium carbonate;
 - [f] adding acid to the organic rich liquid-1 as obtained in step [e] till pH is ca. 4 and separating the organic rich liquid-2 while precipitating gypsum;
 - [g] evaporating the organic rich liquid-2 obtained in step [f] till the solid concentration reaches 50-60% to obtain desalted organics suitable for cattle feed formulation;
 - 15 [h] calcining the precipitated calcium carbonate from step [e] to produce calcium oxide & carbon dioxide;
 - [i] recycling the calcium oxide obtained in step [h] in step [i];
 - [j] recycling the carbon dioxide from step [h] in step [d].
- 20 2. The process as claimed in claim 1, wherein lime used is selected from calcium oxide or calcium hydroxide.
3. The process as claimed in claim 1, wherein the acid used in step [f] is selected from sulphuric acid or phosphoric acid.
4. The process as claimed in claim 1, wherein potassium content in de-salted organics is less than 3% on dry matter basis.
- 25 5. The process as claimed in claim 1, wherein raw material consumption is minimized through calcination of precipitated calcium carbonate and recycling the resultant calcium oxide & carbon dioxide.
6. The process as claimed in claim 1, wherein the desalted organics obtained in step [g] are useful in the preparation of cattle feed formulation.
- 30

**Fig. 1**

[1/1]

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IN2019/050837

A. CLASSIFICATION OF SUBJECT MATTER
C02F1/00 Version=2020.01

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)

C02F

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)

TotalPatent One, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2018/154607 (COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH) 30 August 2018 (30/08/2018) Examples 1-8, Claims 1-9 -----	1-6
A	WO 2016/059651 A1 (COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH) 21 April 2016 (21/04/2016) Examples 1-10, Claims 1-4	1-6

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT
Information on patent family members

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WO 2018/154607 A1	30-08-2018	US 2020010376 A1	09-01-2020
WO 2016/059651 A1	21-04-2016	US 2018230065 A1	16-08-2018
		WO 2016059651 A9	19-05-2016