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(54) Title: A PROCESS FOR PREPARING CRUDE BIO-OIL FROM FEEDSTOCK

(57) Abstract: The present disclosure relates to a process for the conversion of a feedstock to crude bio-oil in the presence of metal nano-particles as catalyst. The process comprising i. charging a mixture, comprising a slurry of 5% to 50 % of a feedstock and a metal compound; ii. stirring the charged mixture to obtain a stirred mixture comprising feedstock and metal nano-particles; iii. introducing hydrogen gas in to the reactor to attain a pressure in the range of 35 to 50 bar; and iv. hydrothermally liquefying the feedstock at a predetermined temperature and at a predetermined pressure of hydrogen for a first predetermined time period in the presence of metal nano-particles to obtain the crude bio-oil.



A PROCESS FOR PREPARING CRUDE BIO-OIL FROM FEEDSTOCK

FIELD

The present disclosure is related to a process for preparing crude bio-oil (CBO) from feedstock.

5

DEFINITIONS

As used in the present disclosure, the following words and phrases are generally intended to have the meaning as set forth below, except to the extent that the context in which they are used indicate otherwise.

- 10 The term “feedstock” in the context of the present disclosure includes, but is not limited to, micro-algae, macro-algae, lipid containing bio-mass, algal bio-mass, bio-mass from wastes such as municipal waste, bio-refinery waste, food processing waste, animal waste, industrial waste, organic waste, urban refuse, wood, agricultural crops or wastes and the like, which can be used as a source of fuel or energy.
- 15 The term “crude bio-oil” in the context of the present disclosure includes, but is not limited to, petroleum crude containing at least one organic product such as free fatty acids, nitrogen containing heterocyclic compounds, polycyclic aromatics, unsaturated compounds and other heavier organic compounds.

20 **BACKGROUND**

Resources of natural fuel are limited and there is a search for viable alternatives. Crude bio-oil (CBO) is a promising alternative to natural fuel. Conventional processes used to obtain crude bio-oil, includes hydrothermal treatment of feedstock.

- 25 Various processes or methods have been suggested to convert a feedstock into bio-fuel in the presence of a catalyst, but the conversion rate is as low as 40 %.

Further, these processes have limitations like extensive waste generation, high energy requirements and high cost.

Therefore, there is felt a need for a cost-effective process that is capable of converting the feedstock into bio-fuel in higher yield.

OBJECTS

- 5 Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows.

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

- 10 An object of the present disclosure is to provide a process for the conversion of feedstock into crude bio-oil with a relatively high yield.

Another object of the present disclosure is to provide a process for the conversion of a cheap and readily available feedstock into crude bio-oil.

Still another object of the present disclosure is to provide a process for the conversion of a feedstock into crude bio-oil in which the catalyst is generated *in-situ*.

- 15 Other objects and advantages of the present disclosure will be more apparent from the following description and examples which are not intended to limit the scope of the present disclosure.

SUMMARY

- 20 The present disclosure provides a process for preparing crude bio-oil from feedstock, said process comprising;
- a. providing in a reactor, a mixture of an aqueous slurry comprising 5% to 50% of said feedstock and metal nano-particles followed by introducing hydrogen gas in said reactor to attain a predetermined pressure;
 - 25 b. hydrothermally liquefying said feedstock in the presence of metal nano-particles at a predetermined temperature for a first predetermined time period to obtain a mixture containing the crude bio-oil; and

- c. purifying the mixture containing the crude bio-oil to obtain a purified crude bio-oil.

The metal nano-particles used for the process are obtained from a metal compound either by *in-situ* generation or the metal nano-particles are prepared separately and added in step 5 (a) to form the mixture of aqueous layer. The size of metal nano-particles produced in the present disclosure, is in the range of 5 to 100 nm.

In accordance with one aspect of the process of the present disclosure, a mixture comprising an aqueous slurry of a feedstock and a metal compound is charged into a reactor. The mixture is then stirred to obtain a slurry comprising metal nano-particles and 10 the feedstock. The slurry is subjected to hydrothermal liquefaction process at a predetermined temperature and at a predetermined pressure of hydrogen for a first predetermined time period to obtain crude bio-oil.

The metal nano-particles of the present disclosure are nano-particles of a metal selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, 15 palladium, ruthenium and rhodium

The process of the present disclosure provides crude bio-oil in a yield 11 to 21% higher than that carried out in the absence of a catalyst.

DETAILED DESCRIPTION

20 The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be 25 practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The description of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or

adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein
5 is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

This invention is not based on a biological resource but on a value added product, and the
10 Crude bio-oil (CBO), which is the subject of this invention, is a value added product, and the original biomass such as algae is not recognizable and is not physically separable from the final product.

The disclosure will now be described with reference to the accompanying embodiments which do not limit the scope and ambit of the disclosure. The description provided is
15 purely by way of example and illustration.

The present disclosure envisages a process for the preparation of crude bio-oil from feedstock.

In accordance with one aspect of the present disclosure, there is provided a process for the preparation of crude bio-oil from feedstock in the presence of metal nano-particles that
20 acts as a catalyst. The process comprises the following steps.

In one embodiment of the process, a reactor is charged with a mixture of an aqueous slurry comprising 5% to 50% of a feedstock and a metal compound and then heated to a temperature in the range of 45 °C to 105 °C with continuous stirring at a predetermined speed for a predetermined time period to obtain metal nano-particles. The metal nano-
25 particles used in the present disclosure can also be prepared separately and then added to the feedstock either during the formation of slurry or thereafter. During the course of stirring, the metal compound is converted into metal nano-particles. After stirring, hydrogen gas is passed in to the reactor to attain a predetermine pressure and then the resultant mixture is heated to a predetermine temperature. The mixture thus formed, is
30 maintained under the same temperature for a first predetermined time period for hydrothermally liquefying the feedstock in the presence of metal nano-particles. The

hydrothermal liquefaction is a complex process comprising different chemical reactions such as hydrolysis, degradation and repolymerization that result in the formation of crude bio-oil from feedstock. During the hydrothermal liquefaction, the carbohydrates, proteins and lipids present in the feedstock react in the presence of metal nano-particles and form crude bio-oil.

The metal nano-particles used as catalyst in hydrothermal liquefaction, reduce the loss of carbon in the form of carbon dioxide or carbon monoxide and result in a higher yield of crude bio-oil.

Further, Metal nanoparticles of the present disclosure help in rupturing the algal cell wall to release its components. The feedstock used in the present disclosure results in oil, ash and water during the course of hydrothermal liquefaction process.

After the hydrothermal liquefaction of the feedstock, the reactor is cooled and the mixture containing the crude bio-oil is collected. The mixture containing the crude bio-oil is purified to obtain purified crude bio-oil.

For purification, the mixture containing the crude bio-oil is filtered and the liquids thus obtained are separated by gravimetric method to obtain purified crude bio-oil.

In accordance with one embodiment of the present disclosure, the feedstock is at least one selected from the group consisting of organic waste, agricultural residues, urban refuse, land and water based plant materials, and microorganisms.

The feedstock used in the present disclosure may also include at least one algae selected from the group consisting of rhodophyta, chlorophyta, phaeophyta, chrysophyta, cryptophyta, dinophyta, tribophyta, glaucophyta, spirulina, nannochloropsis, chlorella, euglena, microcystis, dictyosphaerium anabaena, nodularia, oscillatoria, filamentous algae, spirogyra, hydrodictyon, chara, nitella, oedogonium and phormidium.

In accordance with one exemplary embodiment of the present disclosure, the algae is spirulina.

The spirulina algae used in the present disclosure is native to the Indian continent and procured from Jamnagar, Gujarat and Alibagh, Maharashtra.

In accordance with second exemplary embodiment of the present disclosure, the algae is nannochloropsis.

The nannochloropsis algae used in the present disclosure, was purchased from Solix Biosystems, Inc. USA.

- 5 In accordance with the present disclosure, the size of the metal nano-particles is in the range of 5 nm to 100 nm.

In accordance with the present disclosure, the predetermined temperature for the hydrothermal liquefaction process is in the range of 200 °C to 400 °C.

- 10 In accordance with the present disclosure, the predetermined pressure for hydrogen is in the range of 35 bar to 50 bar.

In accordance with the present disclosure, the first predetermined time period for the hydrothermal liquefaction process is in the range of 10 minutes to 180 minutes.

In accordance with the present disclosure, the ratio of the amount of metal nano-particles and the amount of feedstock ranges from 1:1000 to 1:10.

- 15 The ratio of the amount of metal nano-particles and the amount of feedstock used in the present disclosure, is preferably in the range of 1:1000 to 1:100.

The metal nano-particles of the present disclosure are nano-particles of at least one metal selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.

- 20 In accordance with one embodiment of the present disclosure, the metal nano-particles are prepared *in-situ*.

For *in-situ* preparation of the metal nano-particles, a metal compound is added to the feedstock followed by stirring of the resultant mixture at a speed of 400 rpm to 600 rpm to form an aqueous slurry.

- 25 In accordance with the present disclosure, the metal compound is at least one selected from the group consisting of a metal salt and a metal oxide.

The metal salt of the present disclosure is a salt of at least one metal selected from the group of metals consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.

5 The metal oxide of the present disclosure is an oxide of at least one metal selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.

In accordance with one exemplary embodiment of the present disclosure, the metal salt used is Rhodium chloride.

10 In accordance with the present disclosure, the hydrothermal liquefaction process is carried out for a time period in the range of 10 to 180 minutes and typically 30 to 90 minutes.

Further, the metal nano-particles of the present disclosure can be prepared separately and then added to the aqueous slurry.

In accordance with the present disclosure, the metal nano-particles are supported metal nano-particles.

15 In accordance with the present disclosure, the support used for the metal nano-particles is selected from the group consisting of silica, alumina, zirconia, zeolite, mesoporous support and nano porous support.

In accordance with the present disclosure, the support is in a form selected from the group consisting of extrudates, spheres, pellets and powder.

20 The yield of crude bio-oil and the carbon content in crude bio-oil depends on various factors such as the type of the feedstock, parameters employed during hydrothermal liquefaction, and the metal nano-particles used.

In accordance with the present disclosure, the yield of crude bio-oil is increased by 11% to 21% using the process of the present disclosure.

25 The present disclosure is further described in light of the following experiments which are set forth for illustration purpose only and not to be construed for limiting the scope of the present disclosure. These laboratory scale experiments provided herein can be scaled up to industrial or commercial scale.

EXPERIMENT

A reactor was charged with a slurry comprising 23 g Spirulina algae and 95 g water with 20 mg of rhodium chloride to obtain 0.1 wt % metal nano-particles with respect to the Spirulina algae. This mixture, comprising Spirulina algae and rhodium chloride, was heated at a temperature of 65 °C and then stirred at 500 rpm for 1 hour to obtain a mixture comprising Spirulina algae and rhodium nano-particles.

Hydrogen gas was passed in to the reactor to attain a pressure of 35 bar and the mixture thus formed was heated to a temperature of 350°C. The resultant mixture was maintained under the same temperature condition for 30 min for hydrothermally liquefying the Spirulina algae in the presence of rhodium nano-particles.

The reactor was then cooled and the mixture was collected in a beaker. The mixture was filtered to separate solid portion and liquid portion (containing oil and aqueous phase). The solid portion was washed with dichloromethane and water, and then dried. The oil and aqueous phases present in the liquid portion were separated by gravimetric method. The oil phase, the aqueous phase and the solid portion were separated and measured individually. The oil phase contains crude bio-oil.

The above experiment was repeated in the presence of different metal nano-particles. The results obtained are shown here in **Table 1**.

Table 1: Crude bio-oil yield using Spirulina algae in the presence of various metal nano-particles

Sr. No.	Metal Nanoparticles	CBO Yield, %	% Increase in CBO Yield
1.	Absent	48	
2.	Silver	54	13
3.	Platinum	54	13
4.	Rhodium	55	15
5.	Copper	55	15
6.	Ruthenium	58	21

From **Table 1**, it is observed that the process of the present disclosure (entries 2-6) provides crude bio-oil from Spirulina algae in a yield 13% to 21% higher compared to the process that was carried out in the absence metal nano-particles (entry 1). Further, the increase in % yield was highest in the case of ruthenium nanoparticles.

- 5 Further, the above experiment was repeated using Nannochloropsis algae as feedstock with different metal nano-particles under the same temperature and pressure conditions used for Spirulina algae. The results, thus obtained, are shown here in **Table 2**.

Table 2: Crude bio-oil yield using Nannochloropsis algae as a feedstock

Sr. No.	Metal Nanoparticles	CBO Yield, %	% Increase in CBO Yield
1.	Absent	57	
2.	Rhodium	63	11
3.	Silver	64	13

- 10 From **Table 2**, it is observed that the process of present disclosure (entries 2 and 3) provides crude bio-oil from Nannochloropsis algae in a yield 11% to 13% higher than that carried out in the absence of metal nano-particles.

Thus, the process of the present disclosure provides the crude bio-oil from cheap and readily available feedstock such as Spirulina algae and Nannochloropsis algae thereby
15 making the process of the present disclosure economically viable.

TECHNICAL ADVANCES AND ECONOMICAL SIGNIFICANCE

The present disclosure described herein above has several technical advantages including, but not limited to, the realization of:

- higher yields of crude bio-oil;
- 20 – use of cheap and readily available feedstock for preparing the crude bio-oil;
- use of in-situ generated metal nano-particles as a catalyst;and
- lower energy requirements.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

- 5 The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part
10 of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the
15 numerical values assigned to the parameters, dimensions or quantities fall within the scope of the disclosure, unless there is a statement in the specification specific to the contrary.

While considerable emphasis has been placed herein on the components and component parts of the preferred embodiments, it will be appreciated that many embodiments can be made and that many changes can be made in the preferred embodiments without departing
20 from the principles of the disclosure. These and other changes in the preferred embodiment as well as other embodiments of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

CLAIMS

1. A process for preparing crude bio-oil from feedstock; said process comprising:
 - a. providing in a reactor, a mixture of an aqueous slurry comprising 5% to 50% of said feedstock and metal nano-particles followed by introducing hydrogen
5 gas in said reactor to attain a predetermined pressure;
 - b. hydrothermally liquefying said feedstock in the presence of metal nano-particles at a predetermined temperature for a first predetermined time period to obtain a mixture containing the crude bio-oil; and
 - c. purifying the mixture containing the crude bio-oil to obtain a purified crude
10 bio-oil.
2. The process as claimed in claim 1, wherein said feedstock is at least one selected from the group consisting of organic waste, agricultural residues, urban refuse, land
15 and water based plant materials, and microorganisms.
3. The process as claimed in claim 1, wherein said feedstock is at least one algae selected from the group consisting of rhodophyta, chlorophyta, phaeophyta, chrysophyta, cryptophyta, dinophyta, tribophyta, glaucophyta, spirulina, nannochloropsis, chlorella, euglena, microcystis, dictyosphaerium anabaena,
20 nodularia, oscillatoria, filamentous algae, spirogyra, hydrodictyon, chara, nitella, oedogonium and phormidium.
4. The process as claimed in claim 1, wherein the particle size of metal nano-particles
25 is in the range of 5 nm to 100 nm.
5. The process as claimed in claim 1, wherein said predetermined temperature is in the range of 200 °C to 400°C.
- 30 6. The process as claimed in claim 1, wherein said predetermined pressure is in the range of 35 bar to 50 bar.

7. The process as claimed in claim 1, wherein said first predetermined time period is in the range of 10 minutes to 180 minutes.
- 5 8. The process as claimed in claim 1, wherein the ratio of the amount of said metal nano-particles and the amount of said feedstock is in the range of 1:1000 to 1:10.
9. The process as claimed in claim 1, wherein the ratio of the amount of said metal nano-particles and the amount of said feedstock is in the range of 1:1000 to 1:100.
- 10
10. The process as claimed in claim 1, wherein said metal nano-particles are metal nano-particles of at least one metal selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.
- 15
11. The process as claimed in claim 1, wherein said metal nano-particles are prepared *in-situ* by adding at least one metal compound to said aqueous slurry followed by stirring the resultant mixture at a predetermined speed for a second predetermined time period.
- 20
12. The process as claimed in claim 11, wherein the metal compound is at least one selected from the group consisting of metal salt and metal oxide.
- 25
13. The process as claimed in claim 12, wherein the metal of said metal salt is at least one selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.
- 30
14. The process as claimed in claim 12, wherein the metal of said metal oxide is at least one selected from the group consisting of nickel, molybdenum, cobalt, silver, gold, niobium, platinum, palladium, ruthenium and rhodium.

15. The process as claimed in claim 11, wherein said predetermined speed is in the range of 400 rpm to 600 rpm.

5 16. The process as claimed in claim 11, wherein said second predetermined time period is in the range of 30 minutes to 90 minutes.

10 17. The process as claimed in claim 1, wherein said metal nano-particles are added to the aqueous slurry.

18. The process as claimed in claim 17, wherein said metal nano-particles are supported on at least one support selected from the group consisting of silica, alumina, zirconia, zeolite, mesoporous support and nano porous support; wherein said support is in the form selected from the group consisting of extrudates, spheres, pellets and powder.

15

19. The process as claimed in claim 1, wherein the yield of the crude bio-oil is 11% to 21% higher as compared to the process carried out in the absence of metal nano-particles as claimed in claim 1.

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER
C07C1/00 Version=2015.01

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)

C07C, C10G, C10L, B01J.

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)

PATSEER, ESPACENET, PATENTSCOPE. Search terms: bio fuel, bio oil, bio feed, hydrothermal treatment of biomass, metal nanoparticles, metal oxides.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO2011146277 (A2) LUPTON FRANCIS S[US] ET.AL 24-11-2011 24 NOVEMBER 2011. ABSTRACT, CLAIMS 1-10	1-19
Y	WO2012082367 (A1) ROBERTS VIRGINIA M[US] ET.AL 21-06-2012 21 JUNE 2012 CLAIMS 1-20	1-19
Y	CN101805629A JINSHENG GAO ETAL 18-08-2010 18 AUGUST 2010 ABSTRACT	1-19

☐ 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

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
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WO 2011146277 A2	24-11-2011	US 20110287503 A1	24-11-2011
		EP 2571959 A2	27-03-2013
		AU 2011256474 A1	26-07-2012
WO 2012082367 A1	21-06-2012	US 20120094879 A1	19-04-2012
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		CN 103261378 B	07-10-2015
CN 101805629 A	18-08-2010	AE NONE	