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(54) Title of the invention : SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF

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Filing Date :NA

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(57) Abstract :
The present invention relates to a silicon-free surface-modifier poly(stearyl methacrylate) composition for hydrophobicity comprising Cellulosic fibers composed of tissue papers, pomelo peel: glyoxal (GLA): polyvinyl alcohol (PVA): poly(stearyl methacrylate) (PSMA) in a ratio of 0.08:0.16:.5:1:0.2. A method for synthesis of the composition comprises of mixing pomelo peel and tissue paper in water to obtain a cellulosic pulp, mixing Polyvinyl Alcohol in distilled water to obtain a sol-gel, adding glyoxal to sol-gel obtained in step b), adding Hydrochloric acid to sol-gel obtained in step c) to maintain the pH at 3, mixing obtained solutions of step a) and step d) to get a homogeneous sol-gel, ageing obtained sol-gel (step e) followed by freezing, sublimating freezed sol-gel into waste cellulosic biomass-based aerogel, mixing PSMA into diethyl ether, forming a homogeneous mixture, adding waste cellulosic biomass-based aerogel in homogeneous mixture to obtain a solvent, and evaporating solvent to obtain PSMA-modified hydrophobic aerogel.

No. of Pages : 20 No. of Claims : 5

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Government of India

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Application Details	
APPLICATION NUMBER	202211052362
APPLICATION TYPE	ORDINARY APPLICATION
DATE OF FILING	13/09/2022
APPLICANT NAME	**DIT UNIVERSITY**
TITLE OF INVENTION	"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"
FIELD OF INVENTION	POLYMER TECHNOLOGY
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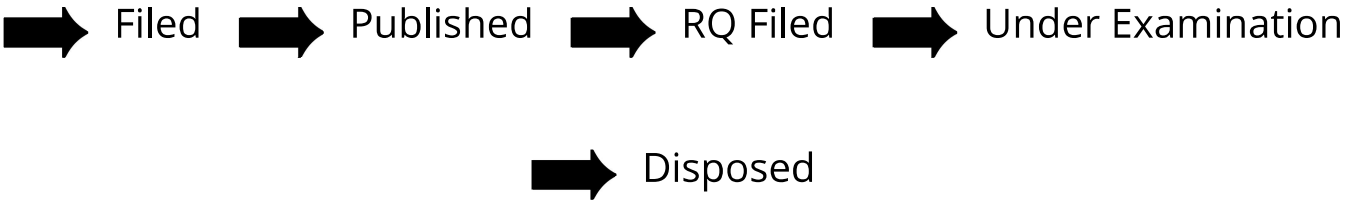
ADDITIONAL-EMAIL (As Per Record)	CONTACT@ELPISANALYTIX.COM
E-MAIL (UPDATED Online)	
PRIORITY DATE	
REQUEST FOR EXAMINATION DATE	--
PUBLICATION DATE (U/S 11A)	23/09/2022

Application Status	
APPLICATION STATUS	**Awaiting Request for Examination**

View Documents



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Document Name	*Created Date/Uploaded Date
202211052362-FORM-9 [14-09-2022(online)].pdf	14/09/2022
202211052362-COMPLETE SPECIFICATION [13-09-2022(online)].pdf	13/09/2022
202211052362-DECLARATION OF INVENTORSHIP (FORM 5) [13-09-2022(online)].pdf	13/09/2022
202211052362-DRAWINGS [13-09-2022(online)].pdf	13/09/2022
202211052362-EDUCATIONAL INSTITUTION(S) [13-09-2022(online)].pdf	13/09/2022
202211052362-EVIDENCE FOR REGISTRATION UNDER SSI [13-09-2022(online)].pdf	13/09/2022
202211052362-EVIDENCE FOR REGISTRATION UNDER SSI(FORM-28) [13-09-2022(online)].pdf	13/09/2022
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202211052362-POWER OF AUTHORITY [13-09-2022(online)].pdf	13/09/2022
202211052362-STATEMENT OF UNDERTAKING (FORM 3) [13-09-2022(online)].pdf	13/09/2022

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FORM 1 THE PATENTS ACT 1970 (39 OF 1970) and THE PATENTS RULES, 2003 **APPLICATION FOR GRANT OF PATENT** (See section 7, 54 and 135 and rule sub rule (1) of rule 20)	(FOR OFFICE USE ONLY)	
	Application No.:	
	Filing Date:	
	Amount of Fee Paid:	
	CBR No:	
	Signature:	

1. **APPLICANT'S REFERENCE / IDENTIFICATION NO. (AS ALLOTED BY THE OFFICE)**	

2. **TYPE OF APPLICATION**

Ordinary (✓)		Convention ( )		PCT – NP ( )	
Divisional ( )	Patent of Addition ( )	Divisional ( )	Patent of Addition ( )	Divisional ( )	Patent of Addition ( )

3 A. APPLICANT(S)

Name in Full	Nationality	Country of Residence	Address of the Applicant	
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			Street	Mussoorie Diversion Road, Village Makkawala, Dehradun, Uttarakhand, India
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			State	Uttarakhand
			Country	India
			Pin Code	248001

3 B. CATEGORY OF APPLICANT

Natural Person ()	Other than Natural Person		
	Small Entity ( )	Startup ( )	Other (✓)

4. INVENTOR(S)

Are all the inventor(s) same as the applicant(s) named above?	Yes ()	No (✓)

If "No" furnish the details of the inventor(s)

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			Country	India
			Pin Code	248009

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			State	Uttarakhand
			Country	India
			Pin Code	248009

Name in Full	Nationality	Country of Residence	Address of the Inventor	
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			Street	Associate professor, Department of Chemical Engineering, GMR Institute of Technology
			City	
			State	Andhra Pradesh
			Country	India
			Pin Code	532127

5. TITLE OF THE INVENTION -

"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"

6. AUTHORISED REGISTERED PATENT AGENT(S)	INPA NO.	2969
	Name	Ms. Ojeswini Bondalapati
	Mobile No.:	+91 7382714119

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	Telephone No.	NA
	Mobile No.	+91 9582649699
	Fax No.	NA
	Email ID	contact@elpisanalytix.com

8. IN CASE OF APPLICATION CLAIMING PRIORITY OF APPLICATION FILED IN CONVENTION COUNTRY, PARTICULARS OF CONVENTION APPLICATION

Country	Application Number	Filing Date	Name of the Applicant	Title of the Invention	IPC (as classified in the convention country
NA	NA	NA	NA	NA	NA

9. IN CASE OF PCT NATIONAL PHASE, PARTICULARS OF INTERNATIONAL APPLICATION FILED UNDER PATENT CO-OPERATION TREATY (PCT)

International Application Number	International filing date
NA	NA

10. IN CASE OF DIVISIONAL APPLICATION FILED UNDER SECTION 16, PARTICULARS OF ORIGINAL (FIRST) APPLICATION

Original (first) application no.	Date of filing of Original (first) application
NA	NA

11. IN CASE OF PATENT OF ADDITION FILED UNDER SECTION 54, PARTICULARS OF MAIN APPLICATION OR PATENT

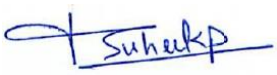
Main application/patent no.	Date of filing of main application
NA	NA

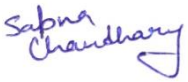
12. DECLARATIONS

(i) Declaration by the inventor(s):

I, the above-named inventor is the true & first inventor for this invention and declare that the applicant herein is my assignee or legal representative.

a) Date: 9th day of September, 2022

1. Signature: 
Name: Suheel Kumar Porwal

2. Signature: 
Name: Sapna Chaudhary

3. Signature: 
Name: Tapas K Dora

(ii) Declaration by the applicant(s) in the convention country

I, the applicant in the convention country declare that the applicant herein is my assignee or legal representative.

a) Date: _____ NA _____
b) Signature: _____ NA _____
c) Name of the signatory: NA

(iii) Declaration by the applicant:

I/We, the applicant(s) hereby declare(s) that:-

[✓] I am in possession of the above mentioned invention.

[✓] The complete specification relating to the invention is filled with this application.

[ ] The invention as disclosed in the specification uses the biological material from India and the necessary permission from the competent authority shall be submitted by me before the grant of patent to me.

[✓] There is no lawful ground of objection to the grant of patent to me.

[ ] I am the true and first Inventor.

[✓] I am the assignee or legal representative of true & first inventor.

[ ] The application or each of the applications, particulars of which are given in Paragraph-8 was the first application in convention country/countries in respect of my invention.

[ ] I claim the priority from the above mentioned application(s) filed in convention country/countries and state that no application for protection in respect of invention had been made in a convention country before that date by me or by any person from which I derive the title.

[ ] My application in India is based on International Application under Patent Cooperation Treaty (PCT) as mentioned in Paragraph-9.

[ ] The application is divided out of my application particulars of which is given in paragraph-10 and pray that this application may be treated as deemed to have been filed on _____under section 16 of the Act.

[ ] The said invention is an improvement in or modification of the invention particulars of which are given in Paragraph-11.

13. FOLLOWING ARE THE ATTACHMENTS WITH THE APPLICATION

a) Form 2

Item	Details	Fee	Remarks
Complete Specification	No. of Pages: 13	--	--
No. of Claim(s)	No. of Claims: 05 and No. of Pages: 02	--	--
Abstract	No. of Page: 01	--	--
Drawing(s)	No. of Drawings: 07 and No. of Pages: 04	--	--

b) Complete specification (in confirmation with the international application)/as amended before the International Preliminary Examination Authority (IPEA), as applicable,
c) Drawings (in confirmation with the international application)/as amended before the International Preliminary Examination Authority (IPEA), as applicable,
d) Statement and undertaking on Form-3,
e) Declaration of Inventorship on Form-5,
f) Copy of International Application Status Report,
g) Copy of Notification of receipt of record copy (PCT/IB/301),
h) Copy of Notification Concerning Submission or Transmittal of Priority Document (PCT/IB/304),
i) Copy of International Search Report,

Deposit of Total Fee_____1600_____

I hereby declare that to the best of my knowledge, information and belief the facts and matters stated herein are correct and I request that a patent may be granted to me for the said invention.

Dated this: 9th day of September, 2022

Ojeswini Bondalapati
AGENT FOR THE APPLICANT
IN/PA/2969

To,
The Controller of Patents,
The Patent Office,
at New Delhi

FORM 5
THE PATENTS ACT 1970
(39 of 1970)
&
THE PATENTS RULES, 2003
DECLARATION AS TO INVENTORSHIP
[See section 10 (6) and 13 (6)]

1. APPLICANT

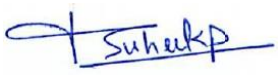
Name	Nationality	Address
DIT University	Indian	Mussoorie Diversion Road, Village Makkawala, Dehradun, Uttarakhand, India -248001

hereby declare that the true and first inventor of the invention disclosed in the complete specification filed in pursuance of our application titled **"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"** is:

2. INVENTOR

Name	Nationality	Address
Suheel Kumar Porwal	Indian	Assistant Professor, Department of Chemistry, DIT University, Dehradun Uttarakhand, 248009
Sapna Chaudhary	Indian	Research Scholar, Department of Chemistry, DIT University, Dehradun, Uttarakhand, 248009
Tapas K Dora	Indian	Associate professor, Department of Chemical Engineering, GMR Institute of Technology, Andhra Pradesh- 532127

Dated this 13th day of September, 2022

1. Signature: 
Name: Suheel Kumar Porwal

2. Signature: 
Name: Sapna Chaudhary

3. Signature: 
Name: Tapas K Dora

Ojeswini Bondalapati
(AGENT FOR THE APPLICANT)
(IN/PA/2969)

To,
The Controller of Patents,
The Patent Office,
at Delhi

Patent Search

Back to search (/PublicSearch/)

Total Document(s): 1

Page: First << 1 >> Last

Application Number	Title	Application Date	Status	
202211052362	"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"	13/09/2022	Published	Application Status

Total Document(s): 1 Page: First << 1 >> Last

Page last updated on: 26/06/2019

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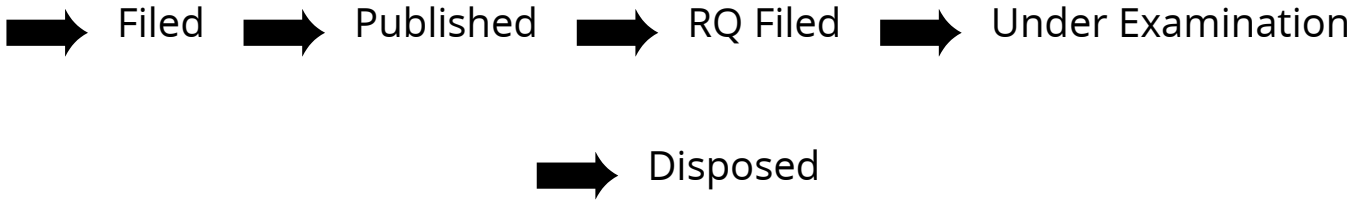
Application Details	
APPLICATION NUMBER	202211052362
APPLICATION TYPE	ORDINARY APPLICATION
DATE OF FILING	13/09/2022
APPLICANT NAME	**DIT UNIVERSITY**
TITLE OF INVENTION	"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"
FIELD OF INVENTION	POLYMER TECHNOLOGY
E-MAIL (As Per Record)	OJESWINI@GMAIL.COM
ADDITIONAL-EMAIL (As Per Record)	CONTACT@ELPISANALYTIX.COM
E-MAIL (UPDATED Online)	
PRIORITY DATE	
REQUEST FOR EXAMINATION DATE	--
PUBLICATION DATE (U/S 11A)	23/09/2022

Application Status	
APPLICATION STATUS	**Awaiting Request for Examination**

View Documents



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FORM 2

THE PATENTS ACT, 1970
(39 of 1970)

&

THE PATENTS RULES, 2003

COMPLETE SPECIFICATION
(See Section 10; rule 13)

Title of the Invention

"SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF"

APPLICANTS:

Name **: DIT University**
Nationality **:** Indian
Address **:** Mussoorie Diversion Road, Village Makkawala, Dehradun, Uttarakhand, India-248009

The following specification particularly describes the invention and the manner in which it is performed.

TECHNICAL FIELD

[0001] The present invention relates to a silicon-free surface-modifier poly (stearyl methacrylate) (SPSM) composition and method for synthesis thereof from a cellulosic biomass comprising pomelo peel and tissue paper for effective oil-water separation.

BACKGROUND ART

[0002] The surface modification of hydrophilic adsorbents is one of the expensive and time-consuming processes as it involves costly silicon-mediated chemicals and chemical vapour deposition methods to get the hydrophobic adsorbent. These hydrophobic adsorbents are frequently utilized to recover oil spillage in water, but their low oil absorption capacity and costly hydrophobic modifications limit applicability worldwide.

[0003] Silicon-free surface-modifier for hydrophobicity in cellulosic aerogels are rarelyreported. The various silicon-mediated surface modifiers, viz. methyltrimethoxysilane (MTMS) and trimethylchlorosilane (TMC) were used to attain hydrophobicity in the cellulosic aerogels via chemical vapour deposition (CVD). However, the CVD method is uneconomical, and the utilization of MTMS and TMC makes it incompatible due to their high cost and toxicity.

[0004] **EP1974784A1** discloses about a filter for oil-water separation comprising water-repellent resin fiber and inorganic fiber. Further, the present invention provides a device for oil-water separation and a method for oil-water separation, using the filter.

[0005] **US20140224733A1** discloses about combination of colloidal silica in conjunction with a chemical, such as a cationically-modified water soluble polymer, e.g. aluminum brine dispersion polymer, clarifies combinations of oil and water, for instance wastewater comprising an oil-in-water emulsion. The colloidal silica may have an average silica particle size of about 0.5 to about 10

nanometers.

[0006] Conventionally, many compositions and methods have developed for treatment for treatment of Gestational diabetes mellitus (GDM). However, such approaches although were effective but were extremely costly which makes them beyond the reach of large group of the society.

[0007] Therefore, there is a need to develop a composition which is economical, non-toxic, eco-friendly and effective for oil recovery by separating oil-water emulsion and moreover, can be established in large scale industries such ad petroleum, refineries etc.

OBJECTS OF THE INVENTION

[0008] The principal object of the present invention is to overcome the disadvantages of the prior art by providing.

[0009] An object of the present invention is to provide a silicon-free surface-modifier poly (stearyl methacrylate) (SPSM) composition and method for synthesis thereof from a cellulosic biomass which is eco-friendly, non-toxic, low-density for effective oil-water separation.

[0010] Another object of the present invention is to provide a composition which provides fast adsorption and quick separation of oil in water.

[0011] Another object of the present invention is to provide a composition through solvent exchange deposition, which does not require any instrument.

[0012] Another object of the present invention is to provide a composition, which is economical.

[0013] The foregoing and other objects of the present invention will become readily apparent upon further review of the following detailed description of the embodiments as illustrated in the accompanying drawings.

SUMMARY OF THE INVENTION

[0014] The present invention relates to a silicon-free surface-modifier poly (stearyl methacrylate) (SPSM) composition and method for synthesis thereof derived from a cellulosic biomass which is eco-friendly, non-toxic, low-density for effective oil-water separation.

[0015] According to an embodiment of the present invention, a silicon-free surface-modifier poly(stearyl methacrylate) composition for hydrophobicity comprising, cellulosic fibers composed of tissue papers, pomelo peel: glyoxal (GLA): polyvinyl alcohol (PVA): poly(stearyl methacrylate) (PSMA) in a ratio of 0.08:0.16:.5:1:0.2.

[0016] According to another embodiment of the present invention, a method for synthesis of the composition comprises of the following steps: pomelo peel and tissue paper are mixed in water at room temperature through stirring to obtain a cellulosic pulp, Polyvinyl Alcohol (PVA) is mixed in distilled water by stirring for 30 mins at 70°C to obtain a sol-gel, glyoxal (GLX) is added to the sol-gel obtained in step b), Hydrochloric acid (HCl) is added to the sol-gel obtained in step c) to maintain the pH at 3, obtained solutions of step a) is mixed with step d) by continuously stirring for a time period in the range of 30 min to 1.5 hr to get a homogeneous sol-gel, the sol-gel (step e) is subjected to aging in an oven at 85°C for 4 hrs followed by freezing at -20°C, the freezed sol-gel is subjected to sublimation into waste cellulosic biomass-based aerogel in a lyophilizer, PSMA is mixed into diethyl ether to from a homogeneous mixture for solvent exchange deposition, the waste cellulosic biomass-based aerogel (obtained in step(g)) is added in the homogeneous mixture obtained in step i) to obtain a solvent, and, the solvent is evaporated at room temperature to obtain PSMA-modified hydrophobic aerogel.

[0017] While the invention has been described and shown with reference to the preferred embodiment, it will be apparent that variations might be possible that

would fall within the scope of the present invention.

BRIEF DESCRIPTION OF DRAWINGS

[0018] So that the manner in which the above-recited features of the present invention can be understood in detail, a more particular description of the invention, briefly summarized above, may have been referred to by embodiments, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only typical embodiments of this invention and are therefore not to be considered limiting of its scope, for the invention may admit to other equally effective embodiments.

[0019] These and other features, benefits, and advantages of the present invention will become apparent by reference to the following text figure, with like reference numbers referring to like structures across the views, wherein:

Figure 1a illustrates a pictorial representation of the macroscopic photographs of the cylindrical aerogel;

Figure 1b illustrates a pictorial representation of the aerogel representing the light nature;

Figure 2 a-c illustrates hydrophobicity of the aerogel by water contact angle (WCA);

Figure 3 illustrates a pictorial representation of the surface morphology of TPPA aerogel examined through scanning electron microscope;

Figure 4 illustrates a graphical representation of FTIR (Fourier transformed infrared) spectrum conducted for TPPA aerogel;

Figure 5 illustrates a graphical representation of the nitrogen adsorption and desorption isotherm conducted for TPPA aerogel;

Figure 6 illustrates graphical representation of aerogel's thermal stability

measured through Thermal Gravimetric Analyser; and

Figure 7 illustrates a pictorial representation of the oil absorption capacity of TPPA.

DETAILED DESCRIPTION OF THE INVENTION

[0020] While the present invention is described herein by way of example using embodiments and illustrative drawings, those skilled in the art will recognize that the invention is not limited to the embodiments of drawing or drawings described and are not intended to represent the scale of the various components. Further, some components that may form a part of the invention may not be illustrated in certain figures, for ease of illustration, and such omissions do not limit the embodiments outlined in any way. It should be understood that the drawings and the detailed description thereto are not intended to limit the invention to the particular form disclosed, but on the contrary, the invention is to cover all modifications, equivalents, and alternatives falling within the scope of the present invention as defined by the appended claim.

[0021] As used throughout this description, the word "may" be used in a permissive sense (i.e. meaning having the potential to), rather than the mandatory sense, (i.e. meaning must). Further, the words "a" or "an" mean "at least one" and the word "plurality" means "one or more" unless otherwise mentioned. Furthermore, the terminology and phraseology used herein are solely used for descriptive purposes and should not be construed as limiting in scope. Language such as "including," "comprising," "having," "containing," or "involving," and variations thereof, is intended to be broad and encompass the subject matter listed thereafter, equivalents, and additional subject matter not recited, and is not intended to exclude other additives, components, integers, or steps. Likewise, the term "comprising" is considered synonymous with the terms "including" or "containing" for applicable legal purposes. Any discussion of document acts, materials, devices, articles, and the like are included in the specification solely for

the purpose of providing a context for the present invention. It is not suggested or represented that any or all these matters form part of the prior art base or were common general knowledge in the field relevant to the present invention.

[0022] In this disclosure, whenever a composition or an element or a group of elements is preceded with the transitional phrase “comprising”, it is understood that we also contemplate the same composition, element, or group of elements with transitional phrases “consisting of”, “consisting”, “selected from the group of consisting of, “including”, or “is” preceding the recitation of the composition, element or group of elements and vice versa.

[0023] The present invention relates to an a silicon-free surface-modifier poly (stearyl methacrylate) (SPSM) composition and method for synthesis thereof from a cellulosic biomass which is economical and effective for recovery of oil.

[0024] According to an embodiment of the present invention, a silicon-free (SPSM) surface-modifier poly(stearyl methacrylate) composition for hydrophobicity comprising, cellulosic fibers composed of tissue papers, pomelo peel: glyoxal (GLA): polyvinyl alcohol (PVA): poly(stearyl methacrylate) (PSMA) in a ratio of 0.08:0.16:.5:1:0.2.

[0025] The poly(stearyl methacrylate) (PSMA) used herein, is used for surface modifications. The Cellulosic fibers composed of tissue papers, pomelo peel, glyoxal (GLA), and polyvinyl alcohol (PVA) are combined to form hydrophobic aerogel (TPPA).

[0026] A method for synthesis of the composition comprising the following steps: mixing pomelo peel and tissue paper in water at room temperature through stirring to obtain a cellulosic pulp, mixing Polyvinyl Alcohol (PVA) in distilled water with stirring for 30 mins at 70°C to obtain a sol-gel, adding glyoxal (GLX) to sol-gel obtained in step b), adding Hydrochloric acid (HCl) to sol-gel obtained in step c) to maintain the pH at 3, mixing obtained solutions of step a) and step d) by continuously stirring for a time period in the range of 30 min to 1.5 hr to get a

homogeneous sol-gel, ageing obtained sol-gel (step e) in an oven at 85°C for 4 hrs followed by freezing at -20°C, sublimating freezed sol-gel into waste cellulosic biomass-based aerogel in a lyophilizer, mixing PSMA into diethyl ether, forming a homogeneous mixture for solvent exchange deposition, adding waste cellulosic biomass-based aerogel (obtained in step(g)) in homogeneous mixture obtained in step i) to obtain a solvent, and evaporating the solvent at room temperature to obtain PSMA-modified hydrophobic aerogel.

[0027] The PSMA modified hydrophobic aerogel synthesized herein, has an oil adsorption capacity of 29.65 g/g.

[0028] EXAMPLE

[0029] According to another embodiment of the present invention, a method for synthesis of the composition comprises of the following steps: 0.5 g of pomelo peel and1.0 g m of tissue paper are mixed in water at room temperature through stirring to obtain a cellulosic pulp, 6 wt% of Polyvinyl Alcohol (PVA) is mixed in distilled water by stirring for 30 mins at 70°C to obtain a sol-gel, 40 wt% of glyoxal (GLX) is added to the sol-gel obtained in step b), Hydrochloric acid (HCl) is added to the sol-gel obtained in step c) to maintain the pH at 3, obtained solutions of step a) is mixed with step d) by continuously stirring for a time period of 1 hr to get a homogeneous sol-gel, the sol-gel (step e) is subjected to aging in an oven at 85°C for 4 hrs followed by freezing at -20°C, the freezed sol-gel is subjected to sublimation into waste cellulosic biomass-based aerogel in a lyophilizer, PSMA is mixed into diethyl ether to from a homogeneous mixture for solvent exchange deposition, the waste cellulosic biomass-based aerogel (obtained in step(g)) is added in the homogeneous mixture obtained in step i) to obtain a solvent, and, the solvent is evaporated at room temperature to obtain PSMA-modified hydrophobic aerogel.

[0030] The synthesis of PSMA-modified hydrophobic aerogel in detail incudes the following steps:

Step I: Processing of Cellulosic waste

[0031] Cellulosic material of waste fruit peel (pomelo peel) was washed with water and dried under sunlight, followed by grinding into powder.

Step II: Hydrophilic aerogel

[0032] Poly (vinyl alcohol) (PVA, 6 wt%) was prepared by dissolving 6g of PVA in hot double distilled water (100 mL) with continuous stirring at 60-70 °C by using the probe sonicator (PRO-650, LAB MAN) for one hour to get a better homogeneous solution. In this solution, the pre-treated powder of pomelo peel (0.5 g) and tissue paper (1.0 g) was added to 50 mL double distilled water to get the pulp and stirring was continued (Tarsons 6040) for 30 min at 500 rpm. Further, glyoxal (GLX, 40 wt%) was added to the above solution in proportions of PP: TP: GLX: PVA in 0.08: 0.16: 0.5:1 ratios to get an effective cross-linking network between PVA and pomelo peel doped tissue paper (PP-TP). The pH~3 was adjusted for effective condensation, and the solution was placed at 80 °C for 4 h. The mixture was transferred into the desired plastic moulds and kept in a deep freezer (Subzero Lab Instruments, Chennai, India) for 3-4 h at -20 °C, then sublimated in a freeze dryer (Subzero Lab Instruments, Chennai, India) at -50 °C and 0.001 mbar pressure for 48 h to obtain the hybrid aerogels as 1:3 ratio named as TPP.

Step II: Hydrophobic aerogel

[0033] Poly(stearyl methacrylate) was prepared by the addition polymerization of Stearyl methacrylate (5.0 g) in the presence of benzoyl peroxide (10 mg) in toluene (2 mL) at 100 ^{o}C for an hour. It was then cooled, and the reaction was terminated by pouring the reaction mixture into hexane. The unreacted starting material was removed by decantation with the hexane and furnished the colourless copolymer in a 97% yield (4.9 g). The synthesized polymer (200 mg) was dissolved in 10 mL diethyl ether, and a hydrophilic adsorbent (300 mg) was dipped. The sample was placed at room temperature for slow evaporation (24 h) to get the surface-modified hydrophobic adsorbent (TPPA).

[0034] The PSMA-modified hydrophobic aerogel is subjected to characterization

studies as mentioned below:

Physical nature:

[0035] Referring to figure 1a, the macroscopic photographs of the cylindrical aerogel (3 cm diameter x 3 cm height). Additionally, the prepared aerogel can stand on a flower petal leaf without bending, demonstrating the ultra-light nature of the materials, as shown in Fig 1b.

Hydrophobicity:

[0036] Referring to figure 2 a-c, hydrophobicity of the aerogel the was examined by water contact angle (WCA) measured using a goniometer system (Rame-hart, Model 90) by applying distilled water or oil. The droplets of coloured water were placed on the surface of the PSMA modified sample (Fig. 2a) and its cross-sectional-cut surface (Fig. 2b); the coloured water drops stood on the surface without penetrating inside, which confirms the hydrophobicity of the entire sample. Finally, the water contact angle of the modified sample was measured and found to be 138 (Fig. 2c), indicating that the PSMA coating might last longer.

Microscopic analysis of pomelo peel/tissue paper aerogel

[0037] Referring to fig.3, The surface morphology of TPPA aerogel was examined by scanning electron microscope (FEI Quanta 200 F SEM). The SEM morphology of the synthesized TPPA is shown at a magnification of 50 um in Fig. 3. The samples performed for microscopic analysis were coated with 2 nm of Au-pd layer to avoid electron charging. The inter-linking of TPPA fibres results in a cabbage-like structure that can withstand loads and preserve its original shape after unloading. The TPP fibres are connected to the PVA chain by acetal bridges, resulting in a 3D porous network that allows the TPPA to be flexible. Pore's nonuniformity can be seen, which is caused by sample cutting before mounting the SEM plate.

FTIR (Fourier transformed infrared) spectrum

[0038] Referring to figure 4, FTIR spectrum was performed to determine the aerogel's functional group using Fourier transform infrared (FTIR) (NICOLET iS50) spectroscopy. The FT-IR spectrum was created by averaging 64 scans with a scan resolution of 4 cm^{-1}. However, PSMA modified hydrophobic aerogel was obtained, which was confirmed by FTIR analysis shown in Fig. 4. The strong band observed at 1729 cm^{-1} corresponds to the ester carbonyl from the PSMA polymer, and the band observed at 2917 cm^{-1}, and 2849 cm^{-1} corresponds to the $–CH_2$ and $–CH_3$, respectively

[0039] Nitrogen adsorption and desorption isotherm

[0040] Referring to figure 5, the nitrogen adsorption and desorption isotherm was performed to determine the specific surface area and average pore diameter (Micrometrics, ASAP 2020). The Brunauer-Emmett-Teller (BET) isotherms for the synthesized TPPA are shown in Fig. 5. The nitrogen adsorption and desorption curves show type-IV isotherm. Furthermore, the separation of the adsorption and desorption isotherms occurred between 0.4 and 0.5 relative pressure, indicating the presence of mesopores in synthesized TPPA. The specific surface area was found to be 12 m^2/g.

[0041] Thermal stability

[0042] Referring to figure 6, the aerogel's thermal stability was analyzed using Thermal Gravimetric Analyser (TGA Q50 V20.13 Build 39) by heating the TPPA at 600 ^oC with a heating rate of 10 °C/min under an air medium. The maximum degradation was observed at 450 ^oC. The TGA analysis revealed decomposition at 155 ^oC, 205 ^oC, 235 ^oC and 400 ^oC corresponding to ~4%, 12%, 18% and 41%. This degradation occurs due to the acetal linkage between PVA and GLX, depolymerization of the polymer and cellulose degradation in the aerogel. The degradation beyond 400 ^oC was observed due to the decomposition of carbonaceous material obtained from cellulose; hence, it is thermally more stable.

[0043] Oil sorption capacity of PSMA modified hydrophobic aerogel

[0044] Referring to fig.7, the oil absorption capacity of TPPA was obtained by using crude oil with a density of 0.81 g/cc. The images were taken during oil sorption testing. As observed, a crossectional cut aerogel was immersed in an oil bath (strained with Sudan Red-G Dye). After oil adsorption, the aerogel floated and changed colour from white to red. The oil adsorption capacity of the TPPA is shown in Fig. 7a. The oil adsorption capacity curve is divided into three sections: rapid, sluggish, and equilibrium. The maximum sorption capacity was observed to be 29.65 g/g. For evidence, the cross-sectionally cut TPPA sample was also placed in the oil-water system. Because of its hydrophobicity, the TPPA could absorb oil (red) in seconds and leave the water, increasing oil/water selectivity.

[0045] The reusability of the TPPA sample was analyzed with 7 consecutive cycles. The TPPA was regenerated by washing with ether and heating to 80 °C in the hot air oven to retrieve the absorbed oil as it reached its maximum adsorption capability. The regenerated sample was carried out for oil adsorption. Even after 7 cycles, the sample has an oil adsorption capacity of 13.42 g/g; as a result, reusability is possible.

[0046] Thus, it is concluded that, a simple, cost-effective and scalable approach has been developed to synthesize silicon-free poly(stearyl methacrylate) for the surface modification of hydrophilic aerogel via solvent exchange deposition. The PSMA-modified aerogel has selectively and effectively removed oil in water. The synthesized material is thermally more stable and used for varying temperature conditions. The hydrophobic aerogel derived from waste cellulosic biomass is a low-cost, reusable, green material that acts as an effective thermal insulator.

[0047] The commercial aspect of the present disclosure is to provide PSMA-modified hydrophobic aerogel derived from waste cellulosic biomass has wide applications in Indian Oil Corporation Limited, Oil and Natural Gas Corporation, Hindustan Petroleum and Reliance Petroleum industries, such as oil spillage in water, oil leakage during transportation, petroleum production operations, waste water treatment and the removal of colouring materials from the textile industries

and are not limited to many other applications.

[0048] Various modifications to these embodiments are apparent to those skilled in the art from the description and the accompanying drawings. The principles associated with the various embodiments described herein may be applied to other embodiments. Therefore, the description is not intended to be limited to the 5 embodiments shown along with the accompanying drawings but is to be providing the broadest scope consistent with the principles and the novel and inventive features disclosed or suggested herein. Accordingly, the invention is anticipated to hold on to all other such alternatives, modifications, and variations that fall within the scope of the present invention and appended claims.

Dated this 13th day of September, 2022

Ojeswini Bondalapati
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CLAIMS

We Claim:

1) A silicon-free surface-modifier poly(stearyl methacrylate) composition for hydrophobicity comprising;

Cellulosic fibers composed of tissue papers, pomelo peel: glyoxal (GLA): polyvinyl alcohol (PVA): poly(stearyl methacrylate) (PSMA) in a ratio of 0.08:0.16:.5:1:0.2

2) The composition as claimed in claim 1, wherein said poly(stearyl methacrylate) (PSMA) is used for surface modifications.

3) The composition as claimed in claim 1, wherein said Cellulosic fibers composed of tissue papers, pomelo peel, glyoxal (GLA), and polyvinyl alcohol (PVA) are combined to form hydrophobic aerogel (TPPA).

4) A method for synthesis of said composition comprising the following steps:

a) mixing said pomelo peel and tissue paper in water at room temperature through stirring to obtain a cellulosic pulp;
b) mixing said Polyvinyl Alcohol (PVA) in distilled water with stirring for 30 mins at 70°C to obtain a sol-gel;
c) adding said glyoxal (GLX) to said sol-gel obtained in step b);
d) adding Hydrochloric acid (HCl) to said sol-gel obtained in step c) to maintain the pH at 3;
e) mixing obtained solutions of said step a) and step d) by continuously stirring for a time period in the range of 30 min to 1.5 hr to get a homogeneous sol-gel;
f) ageing said obtained sol-gel (step e) in an oven at 85°C for 4 hrs followed by freezing at -20°C;
g) sublimating said freezed sol-gel into waste cellulosic biomass-based aerogel in a lyophilizer;

h) mixing said PSMA into diethyl ether, forming a homogeneous mixture for solvent exchange deposition;

i) adding said waste cellulosic biomass-based aerogel (obtained in step(g)) in said homogeneous mixture obtained in step i) to obtain a solvent; and

j) evaporating said solvent at room temperature to obtain PSMA-modified hydrophobic aerogel.

5) The method as claimed in claim 4, wherein said PSMA modified hydrophobic aerogel has an oil adsorption capacity of 29.65 g/g.

Dated this 13th day of September, 2022

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ABSTRACT

“SILICON-FREE SURFACE-MODIFIER POLY (STEARYL METHACRYLATE) COMPOSITION AND METHOD FOR SYNTHESIS THEREOF”

The present invention relates to a silicon-free surface-modifier poly(stearyl methacrylate) composition for hydrophobicity comprising Cellulosic fibers composed of tissue papers, pomelo peel: glyoxal (GLA): polyvinyl alcohol (PVA): poly(stearyl methacrylate) (PSMA) in a ratio of 0.08:0.16:.5:1:0.2. A method for synthesis of the composition comprises of mixing pomelo peel and tissue paper in water to obtain a cellulosic pulp, mixing Polyvinyl Alcohol in distilled water to obtain a sol-gel, adding glyoxal to sol-gel obtained in step b), adding Hydrochloric acid to sol-gel obtained in step c) to maintain the pH at 3, mixing obtained solutions of step a) and step d) to get a homogeneous sol-gel, ageing obtained sol-gel (step e) followed by freezing, sublimating freezed sol-gel into waste cellulosic biomass-based aerogel, mixing PSMA into diethyl ether, forming a homogeneous mixture, adding waste cellulosic biomass-based aerogel in homogeneous mixture to obtain a solvent, and evaporating solvent to obtain PSMA-modified hydrophobic aerogel.