

Final Report

Green Nanocomposites From Fully Renewable Biopolymers : Modified vegetable Oil Reinforced With Rosin Acid And Keratin

(DoRD/Chem Sc/TKM/20-289)

Concise Research Accomplishment:

Modification of epoxidised soybean oil was done successfully by reacting it with methacrylic acid and methacrylic anhydride. • Composites were prepared using modified epoxidised soybean oil with- • Styrene/without styrene • DAPA with three different percentage viz. 20-40 wt% was used as a crosslinker in place of petroleum based compounds. 40 wt% of DAPA showed improved properties. • Composites were prepared with 40 wt% of DAPA and nanoclay (1-5 phr) • Characterisations of the prepared samples were done by ¹H-NMR, FT-IR, XRD, SEM, TEM, TGA. Different properties of composites such as Chemical resistance, Water vapour uptake and LOI were done. • Composites prepared without styrene exhibited better properties compared to those of composites with styrene except the mechanical properties where a marginally improved properties were observed for styrene containing composites. • After successful modification of DAPA composites with 40 wt% of DAPA showed improved properties than the composites with either 20 or 30 wt% of DAPA and DVB 20 wt%. DVB was used for comparison purpose as it contains functionality similar to that of DAPA. • Composites prepared with 40 wt% of DAPA and 3phr nanoclay showed better properties than those of composites having either 1 or 5phr nanoclay. • Work and characterisation of another rosin acid derivative (Trivinyl acrylicpimaric acid, R3) containing higher unsaturation is under progress.

Experimental/ Theoretical Investigation carried out

Modification of epoxidized soybean oil (ESO):

13.2g of ESO was heated and stirred continuously under reflux condition. To this, 4.3g of methacrylic acid, and a pinch of hydroquinone was added. Stirring was initiated and the reaction was carried out for 8 h at 120°C. Excess of methacrylic acid (1.7g) containing hydroquinone was then added and the reaction was allowed to proceed for another 4h keeping the temperature constant. The mixture was cooled to room temperature and purified by extraction in chloroform. It was termed as methacrylated epoxidized soybean oil (MESO). The obtained resin was further modified with methacrylic anhydride. 15.7g of MESO was dissolved in toluene. The mixture was heated and stirred under reflux at 70°C. N-methylimidazole (1wt%) was added as catalyst. 7.7g of methacrylic anhydride containing hydroquinone was added dropwise, to the mixture. The reaction was continued for 3h with stirring. Finally, the mixture was allowed to cool down to room temperature. It is termed as methacrylic anhydride modified epoxidized soybean oil (MAESO).

Preparation of Acrylicpimaric acid (APA):

20g of rosin acid, 5.2 g of acrylic acid and 0.4 g of hydroquinone (as a polymerization inhibitor) were put in a flask equipped with a magnetic stirrer, a reflux condenser. The mixture was stirred at 140 °C for 1 h, 160 °C for 2h and 180 °C for 12 h under inert atmosphere. After that, the reaction was stopped and cooled to room temperature. It was dissolved in anhydrous ether and precipitated in excessive petroleum ether. The precipitate was collected via filtration and washed twice with hot water.

Preparation of Divinyl acrylicpimaric acid (DAPA):

Into a 500 mL three-necked round-bottom flask equipped with a reflux condenser, 5g of APA, 3.5g of potassium carbonate and 4.33ml of acetone were charged. Then a solution of 7g of allyl bromide (0.6 mol) dissolved in 2ml of acetone was added at 50 °C drop by drop. After the mixture was refluxed for 12 h at 70 °C, the reaction was stopped and cooled to room temperature before being washed with dichloromethane (DCM). The organic phase was collected via extraction and washed three times with distilled water before removing solvents in a rotary evaporator at 80 °C. A yellowy coloured product named as DAPA with a viscosity of 5.9 Pa s at 30 °C was obtained. Finally, it was dried in a vacuum oven at 80 °C for 6 h to get the final product.

Preparation of Trivinyl acrylicpimaric acid (R3):

In order to prepare Trivinyl acrylicpimaric acid (R3), Maleopimaric acid (MPA) needs to be prepared. The preparation method for MPA is given below-

Preparation of Maleopimaric acid (MPA):

In a three-necked round bottom flask equipped with a magnetic stirrer and a reflux condenser, rosin acid was taken and reacted with acetic acid. To this mixture, maleic anhydride and p-toluene sulfonic acid (PTS) were added and refluxed. After the reaction was completed, a yellow solid was obtained.

Preparation of Trivinyl acrylicpimaric acid (R3):

In a three-necked round bottom flask equipped with a reflux condenser, MPA was taken and reacted with sodium hydroxide for few hours. Aqueous solution of hydrochloric acid was then added to the system drop by drop. A white precipitate was collected via filtration and washed with water. This intermediate product was termed as MPAC which was dried in the vacuum oven for further reaction. Again in a three-necked round bottom flask MPAC, K₂CO₃ were taken and reacted with a solution of allyl bromide with acetone. The mixture was then refluxed. A yellow viscous liquid termed as R3 was obtained after drying in vacuum oven. ***Preparation of Chicken Feather flour:***

The waste chicken feathers were collected from a local poultry farm Tezpur (India). Waste feathers were brought in the laboratory and washed several times with water followed by laundry detergent and finally with sodium hydroxide to remove blood, manure and extraneous materials. The clean feathers were then dried in a vacuum oven. Dried feathers were chopped and grinded into powder. The grinded samples were sieved through a 70 mesh-sized sieve and kept ready for further experiments.

Formation of composites:

To prepare Methacrylic anhydride modified epoxidised soybean oil (MAESO) based composites, the resin was blended with/without styrene or 20-40wt% of DAPA (with respect to resin). For comparison purpose, DVB (20wt%) a petroleum-based compound, was used with the same MAESO as crosslinker. Composites were also prepared with 1-5wt% of nanoclay/without nanoclay with 40wt% of DAPA respectively. To this, 1wt% of Cobalt (II) 2-ethyl hexanoate solution as a promoter was incorporated and mixed thoroughly. To the mixture, 2wt% of Tert-butylperoxy benzoate (TBP) was added and stirred. 30wt% of grinded chicken feather was added in that resin mixture and hand mixed properly for several minutes. The resin/fiber mixture was then kept at room temperature for 2h. The mixture was

then compressed together via compression molding using a manual hydraulic press and maintaining a temperature and pressure of 140 C and 50 MPa respectively.

Detailed Analysis of result

Biocomposites comprising of polymeric matrix and reinforcing agent from renewable resources have been synthesized successfully. Chicken feather fibers (CF) were used as the reinforcing agent. The Epoxidised soybean oil was modified by reacting with methacrylic acid and methacrylic anhydride. Polymer matrix composites were prepared in a compression mold by using modified epoxidised soybean oil, chicken feather and styrene (crosslinker)/ or without styrene.

1H NMR Study of the Resins:

The NMR studies were used to confirm the formation of resins. The signals of protons for the resins were in accordance with the literature. The peaks appeared in the range 2.8-3.2 ppm was due to epoxy protons while the peaks at 4.0-4.3 ppm and 5.1 ppm were for glyceride backbone of soybean oil. The signal due to epoxy proton almost disappeared. In the 1H-NMR spectrum of MESO resin, the peak appeared at around 4 ppm might be due to -CH-OH groups, whereas the peaks in MAESO spectrum, at 5 ppm were due to -CH-OR group (R is for acetate or methacrylate). The peaks in the range 5.0-6.0 ppm were might be due to the -CH proton of C=C group. (Image attached)

FTIR Analysis of the Resin:

Figure shows the FTIR spectra of ESO, MESO and MAESO respectively. The absorption bands at around 825 cm⁻¹, characteristic of the epoxide group was present in ESO. IR spectrum of MESO, showed double bands at 1739 and 1717 cm⁻¹ representing the carbonyl group whereas peak due to hydroxyl absorption was observed around 3480 cm⁻¹. The intensity of another peak at 1630 cm⁻¹, characteristic of carbon double bond (C=C), was found to increase compared to that of ESO. The peaks appeared in the spectrum of MAESO at 1800 and 1775 cm⁻¹ were for carbonyl group (C=O). The peak assigned for carbon carbon double bond (C=C) appeared at 1636 cm⁻¹ was intense. The peak intensity due to hydroxyl group was very less. The intensity of epoxide peaks was also less compared to that of ESO. All these indicated the successful modification of ESO. (Image attached)

FTIR Analysis of Chicken Feather & Composites prepared with/without styrene:

FTIR spectra of the composite samples confirmed the structural changes of chicken feather fiber resulting from the use of MAESO resin with or without styrene. Chicken feather fiber shows characteristic absorption bands assigned to the peptide bonds (-CONH) as amide A, amide I, amide II and amide III bands. The peaks of chicken feather fiber were in accordance to the literature. An absorption peak corresponding to N-H stretching (amide A) was appeared at 3290 cm⁻¹. For keratin protein, the methyl (CH₃) asymmetric and symmetric modes were observed at 2962 and 2930 cm⁻¹, respectively, while the methylene (CH₂) asymmetric and symmetric modes were found at 2875 and 2855 cm⁻¹. A strong absorption peak at 1652 cm⁻¹ was due to the C=O (amide I). Another peak observed at 1534 cm⁻¹ was assigned to C-N stretching and N-H in-plane bending vibrations (amide II). Also band near 1371 cm⁻¹ assigned to δ(CH) while the weak band at 1238 cm⁻¹ related to the C-N and CO stretching vibrations (amide III) were also noticed. The bands in the wavenumber range of 730-620 cm-

1 were due to alkyl thiols (C-S), originated from amino acid cysteine. In the spectra of both the composites with/without styrene, the peaks at 1739 cm⁻¹ (C=O), 1635 cm⁻¹ (C=C) and 1534 cm⁻¹ (amide II), characteristic for resins and fibers were observed. In the composites the N-H peak intensity was found to decrease as well as shift to lower wavenumber. The shifting of peak was marginally more for composites containing styrene. It was 3280 cm⁻¹ for composites with styrene against 3282 cm⁻¹ for composites without styrene. Further it was seen that the intensity of C=O (amide) peak and amide II peaks decreased. The decrease in peak intensity confirmed the interaction between the feather fiber and the resin. (Image attached)

SEM Study of the composites prepared with/without styrene:

The morphological features of the fractural surface of feather fiber composites were studied by SEM. In the composites prepared with/without styrene, it was observed that there were no voids in the polymer matrix around the fibers indicating better fiber matrix adhesion between the components. The nodes and hooks, as well as the twisted nature of the microfibrills, were responsible for improved adhesion. The fractured surfaces of the styrene free composites appeared smoother than styrene added composites. This suggested that composites without styrene had little bit better fiber-matrix adhesion compared to composites containing styrene.

TGA Study of the composites prepared with/without styrene:

Thermogravimetric analysis for the feather fiber based composites was conducted to identify their degradation characteristics. The percentage weight loss of the sample is recorded when sample is heated, the loss in weight over specific temperature range (room temperature to 600oC) indicates the thermal stability of the sample. In the TGA curve for CF, the weight loss below 100 C was due to removal of moisture. The weight loss occurred in the temperature range 220-400 C was due to breakage of disulphide bonds and elimination of H₂S formed from the cysteine present in CF. The degradation above 400 C was due to partial decomposition of Keratin. The degradation rate of CF based composites was less than that of CF. Further the rate of degradation of composites having no styrene was least. Earlier decomposition of polystyrene chain at higher temperature might be responsible for showing lower thermal stability in CF/Styrene containing composites. Styrene may also acted as a chain extender and reduced the crosslinking density. This facilitated the degradation. The crosslinked density of acrylated epoxidised soybean oil was reported to decrease linearly with the addition of styrene. The composite containing resin and feather showed higher thermal stability than that of composite containing resin, feather and styrene. This might be due to the formation of more rigid network structure between functional groups in resin and feathers in the styrene free composite. (Image attached)

Mechanical Property and Hardness Test of the composites prepared with/without styrene: The tensile and hardness values of MAESO-CFST based composites showed marginally higher value of tensile strength and hardness compared to that of composites without styrene. Chain rigidity and degree of crosslinking are some of the contributing factors in governing the mechanical properties. In this case, the chain contain bulky aromatic ring and is more rigid than MAESO resins which contain mostly long chain fatty acids. The presence of styrene chains in the composites reduced the chain mobility more

than that of composite without styrene. This in turn would enhance the chain stiffness as well as mechanical properties.

Dimensional Stability and Water Vapour Uptake Test of the composites prepared with/without styrene:

Both water vapour uptake and volumetric swelling were found to increase with increase in exposure time. With the increase in time, the capillaries of keratin fibers were getting more time to become filled up with water vapour resulting in an increase in water vapour uptake capacity. Both water vapour uptake and volumetric swelling were less in the case of composites without styrene. The chicken feathers possess both hydrophobic and hydrophilic character at molecular level. In composite prepared without styrene, the percentage of MAESO resin was high which facilitated the wetting as well as adhesion. The improved adhesion between fibers and resin resulted in decrease of water uptake capacity. ***LOI Study of the composites prepared with/without styrene:***

LOI study provides a quantitative measure of flammability of chicken feather based composites. LOI test usually measures the minimum concentration of oxygen required to support the combustion of a sample under specific condition. It was observed that LOI of composites prepared with styrene was lower than that of composites without styrene sample. Effective flame retardant should have some characteristics like localized flame, high char yield, minimal smoke generation etc. Composites without styrene produced small smoke, localized flame than that of composite containing styrene. The early degradation of the polystyrene chain present in the composite might also be responsible for this.

Chemical Resistance Test of the composites prepared with/without styrene:

It was seen that swelling was less in the case of samples without styrene. This might be due to improved crosslinking as explained earlier. It was observed that composite is quite resistive towards acid than alkali solution. The interaction between sodium hydroxide and feather fibers might be responsible for the observed higher swelling of samples in sodium hydroxide compared to acetic acid. The above results showed that composites prepared without styrene exhibited better properties compared to those of composites having styrene except the mechanical properties where a marginally improved properties were observed for styrene containing composites. This suggested that the volatile and hazardous components like styrene can be removed in the preparation of composites. However in order to further improve the properties of the composites a rosin derivative, divinyl acrylicpimaric acid (DAPA) was synthesised from naturally available rosin. This derivative contains two functional double bonds and was used as the crosslinker in the composites preparation. Three different percentage of DAPA i.e., 20-40 wt% and for comparison 20wt% of DVB was employed with respect to the total resin weight in the composites.

¹H-NMR Study of rosin acid and its derivative:

The chemical structures of rosin and Divinyl acrylicpimaric acid (DAPA) were confirmed by ¹H NMR spectra. In the spectrum of DAPA, the peaks appeared in the range of 0.5 -2.5 ppm were attributed to the protons attached on the hydrogen phenanthrene ring. The peaks at ~5.9 ppm, 5.1–5.3 ppm, ~5.4

ppm and ~4.2–4.6 ppm were assigned to the protons Hb and Hc on the terminal –CH=CH₂ groups, proton Ha attached to the unsaturated carbon on the rosin ring and for the allylic hydrogens Hd. .

FTIR Spectra of rosin acid and its derivatives:

The presence of terminal C=C bonds of divinyl acrylicpimaric acid (DAPA) could be confirmed by the characteristic peak appeared at 1655 cm⁻¹, 930 cm⁻¹ and 990 cm⁻¹ which showed proper modification of the rosin acid derivative, DAPA.

FTIR study of the composites prepared with Rosin acid derivative and divinylbenzene:

The decrease in peak intensity and shifting of the peaks confirmed better crosslinking reaction between the MAESO resin, CF and crosslinkers.

Mechanical properties and Hardness Study prepared with Rosin acid derivative and divinylbenzene:

The tensile, flexural and hardness values of the composites were enhanced with increase in the addition of DAPA in the composites. The increase in the amount of DAPA into the composites showed better results than the composite containing DVB (20 wt%). This enhancement in properties was due to increase in interaction between MAESO resin, CF, DAPA/DVB which restricted the movement of polymer chain and as result the mechanical properties and hardness values of composites were significantly improved. This might also be attributed to the hydrogenated phenanthrene ring present in the DAPA which improved the mechanical properties and hardness. Mechanical properties of composite containing DVB were comparable to that of composite containing DAPA.

Thermal study of the composites prepared with Rosin acid derivative and divinylbenzene:

The thermal stability of the chicken feather and composites loaded with various percentages of DAPA and DVB was investigated by the thermogravimetric study. The thermostability of the composites were more than that of chicken feather. It was observed that composites with Divinylbenzene and rosin-based derivative showed almost similar enhancement in thermal stability. With increase in DAPA into the composites, the crosslinking density increased and thermal stability also improved. The residual weight (RW%) was also found to be maximum for composite prepared with highest percentage of DAPA (40 wt%). These results suggested that thermal stability of composites improved due to use of DAPA with MAESO and CF.

Water vapour uptake and Dimensional Stability test of the composites prepared with Rosin acid derivative and divinylbenzene:

The percentage swelling was found to increase with increasing time. With increase in the amount of DAPA both water vapour uptake and volumetric swelling decreased in the composites. Swelling was found to be least in the composite with highest percentage of DAPA. Composites having DAPA showed better results as those of composites containing DVB.

Chemical Resistance test of the composites prepared with Rosin acid derivative and divinylbenzene:

Samples were kept immersed in 4% acetic acid and 4% NaOH solution. With increase in incorporation of DAPA percentage, swelling was decreased. Swelling was found to be least in the composite with highest percentage of DAPA i.e., 40 wt%. This might be due to the better crosslinked structure formed by the DAPA that restricted the path for diffusion of chemicals and hence swelling decreased. It was

observed that composites were quite resistive towards acidic medium than alkali medium. The interaction between sodium hydroxide, CF, DAPA and MAESO resin might be responsible for the higher swelling of the samples in the alkali medium. The above analysis showed that composite containing 40 wt% of DAPA showed improved property than those of composites with 20 and 30 wt% of DAPA and DVB20 wt%. Further, to obtain composites with more improved properties chicken feather fiber was mixed with 40 wt% of DAPA and varying percentage of nanoclay (1-5 phr). FTIR studies of the nanocomposites confirm the changes in chemical structure of chicken feather fiber resulting from the use of crosslinkers with different weight percentage of nanoclay.

Thermal study of the composites prepared by varying percentage of nanoclay:

TGA determines the thermal stability of chicken feather and composites loaded with various percentage of nanoclay. The percentage weight loss of the sample is recorded when sample is heated over specific temperature range (room temperature to 600 C). Initial degradation of chicken feather occurred at 226 C. After incorporation of nanoclay (upto 3 phr) in the composites, the thermal stability of the composites outstandingly improved. The values are maximum when 3wt% of nanoclay was used with the composites. The silicate layers of nanoclay produced a tortuous path for diffusion of the decomposed volatile products throughout the nanocomposite.

Morphological Studies of the composites prepared by varying percentage of nanoclay:

In the micrographs of the composites, the surface of the composites without clay was appeared rough. However the surface roughness was found to decrease with the incorporation of nanoclay. The sample with 3wt% of nanoclay showed that the particles were well spread into the polymer matrix and it changed the morphology of the material with a smooth surface. This indicated an improvement in interaction as well as compatibility among the various components in the composites.

Mechanical properties of the composites prepared by varying percentage of nanoclay:

The tensile, flexural and hardness properties were found to enhance after incorporation of nanoclay to the composites. The values were found to increase up to 3 wt% of nanoclay loading, after that the values decreased. At 5 wt% nanoclay loading composites, the agglomeration of clay took place and as a result, the tensile, flexural and hardness values were found to decrease.

LOI study of the composites prepared by varying percentage of nanoclay:

A significant increase in LOI could be noted when nanoclay was added into the composites. The incorporation of the clay into the composite matrix promoted char formation. The formation of char improved the flame retardancy by insulating the composites. Samples treated with 3 wt% of nanoclay showed higher LOI value. At 5 wt% of nanoclay loading, the agglomeration of clay might have taken place. It was also observed that all the samples produced small black localized flame.

Water Vapour Uptake and Dimensional Stability Test of the composites prepared by varying percentage of nanoclay:

Composite loaded with 3 wt% nanoclay showed least water vapour uptake and highest dimensional stability than the composites having 0, 1 and 5wt% nanoclay. The decreasing trend might be attributed to the presence of silicate layers of clay in the composite which hindered the permeation of water

through the composite. The higher water uptake capacity showed by the composites containing higher percentage of nanoclay (5wt%) was due to agglomeration of clay.

Chemical Resistance Test of the composites prepared by varying percentage of nanoclay:

Samples containing nanoclay swelled less than the sample without nanoclay. Samples having 3wt% of nanoclay swelled least. The above studied suggested that Rosin acid derivative can replace petroleum based substances such as Styrene/dibinylbenzene. Further study was carried out in the preparation of another rosin acid derivative i.e., Trivinyl acrylicpimaric acid (R3), which is expected to produce much better results than the DAPA. Preliminary measurement of composite properties containing trivinyl acrylicpimaric acid (R3):

Chemical resistant and water vapour uptake of R3 containing composite were better than DAPA containing composites. However, thorough investigation is required to confirm the superiority of R3 over DAPA as a crosslinker.

Conclusions

Chicken feather fibre based polymer composites were prepared using methacrylic anhydride epoxidized soybean oil (MAESO) as the resin, chicken feather fibre flour as a reinforcing agent, rosin-based derivative as co-monomer and nanoclay as filler. The modification of epoxidized soybean oil (ESO) was done successfully by reacting with methacrylic acid and methacrylic anhydride to form the MAESO resin. Divinyl acrylicpimaric acid (DAPA) was synthesized successfully as characterized by NMR and FTIR spectroscopy and employed as co-monomer. Three different percentages of nanoclay were loaded into the polymer matrix to investigate the properties of composites. FTIR study indicated the decrease in peak intensities, which confirmed the interaction between chicken feather, resin, DAPA and nanoclay. Composites prepared without styrene exhibited better properties compared to those of composites with styrene except the mechanical properties where a marginally improved properties were observed for styrene containing composites. Composites having 40wt% DAPA exhibited an overall improvement of properties in terms of mechanical, thermal, flame retardancy etc. compared to those of composites containing 20-30wt% of DAPA and DVB (20 wt%). Also composites loaded with 3 wt% of nanoclay with 40 wt% DAPA exhibited an overall improvement of properties in terms of mechanical, thermal, flame retardancy etc. compared to those of composites containing 0, 1 and 5 wt% of nanoclay.

Scope of future work

Work and characterisation of another rosin acid derivative, Trivinyl acrylicpimaric acid (R3) is under progress which is expected to provide much better results as judged by preliminary studies.

Tarunkumar

UP-TO-DATE STATEMENT OF EXPENDITURE

1. SERB Sanction Order No and Date : EMR/2016/000537
2. Name of the PI : Prof. Tarun K. Maji
3. Total project cost : Rs. 23,42,560.00/-
4. Revised project cost :
5. Date of commencement : 26/09/2016
6. Statement of expenditure :

(Month wise expenditure incurred during current financial year)

Month & Year	Expenditure incurred/committed
May 2019	14,000.00
June 2019	36,143.00
July 2019	16,000.00
August 2019	62,420.00
September 2019	86,373.00
October 2019	13,868.00
Total (in Rs.)	2,28,804

1. Grant Received in each year

- a. 1st Year : 11,13,000/-
b. 2nd Year : 5,00,000/-
c. 3rd Year : 6,00,000/-
d. Interest : 61,809/-
e. Total (a+b+c) : 22,74,809/-

Statement of Expenditure

(to be submitted financial year wise i.e. DOS* to 31st March of that financial year 01/04/2019 till 25/09/2019)


Sl. No (I)	Sanction Heads (II)	Total Funds Allocated (indicate sanctioned or revised) (III)	Expenditure Incurred				Total expenditure till 25/09/2019 (VIII=IV+V+VI+VII)	Balance as on 25/09/2019 (IX=III-VIII)	Requirement of funds upto 31 st March 2020	Remarks (If any)
			1 st Year (IV)	2 nd Year (V)	3 rd Year (VI)	3 rd Year (VII) (1/4/2019-25/9/2019)				
1.	Manpower	*9,00,040	62,187	1,73,239	1,68,000	96,010	4,99,436	4,00,604		
2.	Consumable	3,00,000	51,423	1,00,000	1,00,000	41,668	293,091	6,909		
3.	Travel	1,50,000	21,846	16,747	20,732	NIL	59,325	90,675		
4.	Contingencies	1,50,000	25,712	50,000	41,412	20,753	137,877	12,123		
5.	Others, if any (Interest)	61809		NIL	NIL	NIL		61809		
6.	Equipment	5,00,000		4,61,728.25	NIL	NIL	4,61,728.25	38271.75		
7.	Overhead expenses	2,12,960	53,434	NIL	84,850	70,373	208,657	4303		
8.	Total	**22,74,809	2,14,602	8,01,714.25	4,14,994	2,28,804	1,660,114.25	614,694.75	NIL	

* Fund allocated- 10,29,600, Fund received-9,00,040

** Interest added

Taran K. M. Jai
Name and Signature of principal Investigator
(T. K. M. JAI)

Date: Professor
Department of Chemical Sciences
Tezpur University
Tezpur - 784028


Signature of competent Financial Authority
Finance Officer
Tezpur University
Tezpur (Seal)

Date:

* DOS – Date of Start of project

Note:

- Expenditure under the sanctioned heads, at any point of time, should not exceed funds allocated under that head, without prior approval of SERB i.e. Figures in Column (VIII) should not exceed corresponding figures in Column (III)
- Utilization Certificate (Annexure III) for each financial year ending 31st March has to be enclosed along with request for carry forward permission to the next financial year.

**GFR 12 – A [(See Rule 238 (1))]
 UTILIZATION CERTIFICATE (UC) FOR THE YEAR 2019
 in respect of *NON-RECURRING*
 as on 01/04/2019-25/09/2019 to be submitted to SERB
 Is the UC (Provisional/Audited)
 (To be given separately for each financial year ending on 31st March)**

1. Name of the grant receiving Organization : Science and Engineering Research Board (SERB)
2. Name of Principal Investigator(PI) : Prof. Tarun K. Maji
3. SERB Sanction order no. & date : EMR/2016/000537, Dated- 17/09/2016
4. Title of the Project : Green nanocomposite from fully renewable biopolymers: modified vegetable oil reinforced with rosin acid and keratin
5. Name of the SERB Scheme : Core Research Grant (CRG)
6. Whether recurring or non-recurring grants : **NON-RECURRING**
7. Grants position at the beginning of the Financial year
- (i) Carry forward from previous financial year : 38,271.75/-
- (ii) Others, If any : NIL
- (iii) **Total** : **38,271.75/-**

8. Details of grants received, expenditure incurred and closing balances: (Actuals)

Unspent Balance of Grants received previous years [figure as at Sl. No 7(iii)]	Interest Earned thereon	Interest deposited back to the SERB	Grants received during the year	Total Available funds (1+2-3+4)	Expenditure incurred	Closing Balances (5-6)

1	2	3	4			5	6	7
38,271.75/-	-	-	Sanction No (i)	Date (ii)	Amount (iii)	38,271.75/-	NIL	38,271.75/-
			NIL	NIL	NIL			

Component wise utilization of grants:

Grants-in-aid- General	Grant-in-aid-creation for capital assets	Total

Details of grants position at the end of the year

- (i) Balance available at end of financial year : 38,271.75/-
- (ii) Unspent balance refunded to SERB (If any) : 38,271.75/-
- (iii) Balance (Carried forward to next financial year) if applicable : Not applicable

GFR 12 – A [(See Rule
238 (1))]
UTILIZATION CERTIFICATE (UC) FOR THE YEAR 2019
in respect of *NON-RECURRING*
as on 01/04/2019-25/09/2019 to be submitted to SERB
Is the UC (Provisional/Audited)
(To be given separately for each financial year ending on 31st March)

Certified that I have satisfied that the conditions on which grants were sanctioned have been duly fulfilled/are being fulfilled and that I have exercised following checks to see that the money has been actually utilized for the purpose for which it was sanctioned:

- (i) The main accounts and other subsidiary accounts and registers (including assets registers) are maintained as prescribed in the relevant Act/Rules/Standing instructions (mention the Act/Rules) and have been duly audited by designated auditors. The figures depicted above tally with the audited figures mentioned in financial statements/accounts.
- (ii) There exist internal controls for safeguarding public funds/assets, watching outcomes and achievements of physical targets against the financial inputs, ensuring quality in asset creation etc. & the periodic evaluation of internal controls is exercised to ensure their effectiveness.
- (iii) To the best of our knowledge and belief, no transactions have been entered that are in violation of relevant Act/Rules/standing instructions and scheme guidelines.
- (iv) The responsibilities among the key functionaries for execution of the scheme have been assigned in clear terms and are not general in nature.
- (v) The benefits were extended to the intended beneficiaries and only such areas/districts were covered where the scheme was intended to operate.
- (vi) The expenditure on various components of the scheme was in the proportions authorized as per the scheme guidelines and terms and conditions of the grants-in-aid.
- (vii) It has been ensured that the physical and financial performance under **Core Research Grant (CRG)** (Name of the scheme) has been according to the requirements, as prescribed in the guidelines issued by Govt. of India and the performance/targets achieved statement for the year to which the utilization of the fund resulted in outcomes given at Annexure – I duly enclosed.

- (vii) The utilization of the fund resulted in outcomes given at Annexure – II duly enclosed (to be formulated by the Ministry/Department concerned as per their requirements/specifications.)
- (ix) Details of various schemes executed by the agency through grants-in-aid received from the same Ministry or from other Ministries is enclosed at Annexure –II (to be formulated by the Ministry/Department concerned as per their requirements/specifications).

Date:

Place:

<p style="text-align: center; font-size: 1.2em;">Taru Klyji</p> <p>Signature of PI :</p> <p style="text-align: center; font-size: 0.8em;">Professor Department of Chemical Sciences Tezpur University Tezpur - 781026</p>	<p style="text-align: center; font-size: 1.2em;">[Signature]</p> <p>Signature with Seal :</p> <p>Name:</p> <p style="text-align: center; font-size: 0.8em;">Chief Finance Officer Head of University Finance</p>	<p style="text-align: center; font-size: 1.2em;">[Signature]</p> <p>Signature with Seal.....</p> <p>Name:</p> <p style="text-align: center; font-size: 0.8em;">Registrar Head of Organisation</p>
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(Strike out inapplicable terms)

GFR 12 – A
 [(See Rule 238 (1))]
 UTILIZATION CERTIFICATE (UC) FOR THE YEAR 2019
 in respect of *RECURRING*
 as on 01/04/2019-25/09/2019 to be submitted to SERB
 Is the UC (Provisional/Audited)
 (To be given separately for each financial year ending on 31st March)

1. Name of the grant receiving Organization : Science and Engineering Research Board (SERB)
2. Name of Principal Investigator(PI) : Prof. Tarun K. Maji
3. SERB Sanction order no & date : EMR/2016/000537, Dated- 17/09/2016
4. Title of the Project : Green nanocomposite from fully renewable biopolymers: modified vegetable oil reinforced with rosin acid and keratin
5. Name of the SERB Scheme : Core Research Grant (CRG)
6. Whether recurring or non-recurring grants : **RECURRING**
7. Grants position at the beginning of the Financial year

(i)	Carry forward from previous financial year	:	7,96,511/-
(ii)	Others, If any	:	NIL
(iii)	Total	:	7,96,511/-

8. Details of grants received, expenditure incurred and closing balances: (Actuals)

Unspent Balance of Grants received previous years [figure as at Sl. No. 7(iii)]	Interest Earned thereon	Interest deposited back to the SERB	Grants received during the year	Total Available funds (1+2-3+4)	Expenditure incurred	Closing Balances (5-6)
1	2	3	4	5	6	7

7,96,511/-	8,716/-		Sanction No (i)	Date (ii)	Amount (iii)	8,05,227/-	2,28,804/-	5,76,423/-
			NIL	NIL	NIL			

Component wise utilization of grants:

Grants-in-aid- General		Grant-in-aid-creation for capital assets	Total
Manpower Consumable Travel Contingencies	1,58,431/-		2,28,804/-
Overhead Charges	70,373/-		

Details of grants position at the end of the year

- (i) Balance available at end of financial year : 5,76,423/-
- (ii) Unspent balance refunded to SERB (If any) : 5,76,423/-
- (iii) Balance (Carried forward to next financial year) if applicable : Not applicable

GFR 12 – A [(See Rule
238 (1))]
UTILIZATION CERTIFICATE (UC) FOR THE YEAR 2019
in respect of *RECURRING*
as on 01/04/2019-25/09/2019 to be submitted to SERB
Is the UC (*Provisional/Audited*)
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Certified that I have satisfied that the conditions on which grants were sanctioned have been duly fulfilled/are being fulfilled and that I have exercised following checks to see that the money has been actually utilized for the purpose for which it was sanctioned:

- (i) The main accounts and other subsidiary accounts and registers (including assets registers) are maintained as prescribed in the relevant Act/Rules/Standing instructions (mention the Act/Rules) and have been duly audited by designated auditors. The figures depicted above tally with the audited figures mentioned in financial statements/accounts.
- (ii) There exist internal controls for safeguarding public funds/assets, watching outcomes and achievements of physical targets against the financial inputs, ensuring quality in asset creation etc. & the periodic evaluation of internal controls is exercised to ensure their effectiveness.
- (iii) To the best of our knowledge and belief, no transactions have been entered that are in violation of relevant Act/Rules/standing instructions and scheme guidelines.
- (iv) The responsibilities among the key functionaries for execution of the scheme have been assigned in clear terms and are not general in nature.
- (v) The benefits were extended to the intended beneficiaries and only such areas/districts were covered where the scheme was intended to operate.

- (vi) The expenditure on various components of the scheme was in the proportions authorized as per the scheme guidelines and terms and conditions of the grants-in-aid.
- (vii) It has been ensured that the physical and financial performance under **Core Research Grant (CRG)** (Name of the scheme has been according to the requirements, as prescribed in the guidelines issued by Govt. of India and the performance/targets achieved statement for the year to which the utilization of the fund resulted in outcomes given at Annexure – I duly enclosed.
- (viii) The utilization of the fund resulted in outcomes given at Annexure – II duly enclosed (to be formulated by the Ministry/Department concerned as per their requirements/specifications.)
- (ix) Details of various schemes executed by the agency through grants-in-aid received from the same Ministry or from other Ministries is enclosed at Annexure –II (to be formulated by the Ministry/Department concerned as per their requirements/specifications).

Date:

Place:

<p style="text-align: center;"><i>Tarun Khatun</i></p> <p>Signature of PI : Professor</p>	<p style="text-align: center;"><i>[Signature]</i> 17.8.20</p> <p>Signature with Seal :..... Name: Chief Finance Officer (Head of Finance) <i>Tezpur University</i></p>	<p style="text-align: center;"><i>[Signature]</i></p> <p>Signature with Seal..... Name: Registrar Head of Organisation</p>
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Department of Chemical Sciences
Tezpur University
(Strike out inapplicable terms)