

# **PROJECT COMPLETION REPORT**

**Project No: SB/FTP/ETA-88/2013 dated 03.09.2013**

**Title of the project:**

Utilization of the Calcium Carbonate ( $\text{CaCO}_3$ ) obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms.

**Principal Investigator:**

Satadru Kashyap  
Assistant Professor  
Department of Mechanical Engineering  
Tezpur University  
Napaam, Tezpur, Assam  
PIN-784028

**Implementing Institution:**

Tezpur University  
Napaam, Tezpur, Assam  
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Utilization of the Calcium Carbonate ( $\text{CaCO}_3$ ) obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms. (Project No. SB/FTP/ETA-88/2013)

**2. Principal Investigator:**

Satadru Kashyap  
Assistant Professor  
Department of Mechanical Engineering  
Tezpur University  
Napaam, Tezpur, Assam  
PIN-784028

**3. Implementing Institution(s) and other collaborating Institution(s):**

Tezpur University  
Napaam, Tezpur, Assam  
PIN-784028

**4. Date of commencement: 03.09.2013**

**5. Planned date of completion: 02.09.2016**

**6. Actual date of completion: 02.09.2016**

**7. Objectives as stated in the project proposal:**

(a) Calcium Carbonate based lime sludge of paper industry can be used to fabricate 'lime sludge filled polyethylene composites' with comparable mechanical properties than commercially used polyethylene grades. This will generate provisions for (i) utilization of potentially hazardous lime sludge from Indian paper industries and (ii) make the polymer composite economically more viable (as lime sludge would be used a filler material to reduce production cost).

(b) Determination and characterization of mechanical properties of the 'lime sludge filled polyethylene composites'. A model can be developed for investigation, prognosis and prediction of manufacturing parameters and compositional changes required to achieve optimal mechanical properties in lime sludge filled polyethylene composites.

**8. Deviation made from original objectives if any, while implementing the project and reasons thereof:**

In addition to fabrication and characterization of lime sludge filled polyethylene composites, it was also proposed to develop an optimization model which may be used in predicting the properties of lime sludge filled polymeric composites for various weight fractions of filler additions. However, thermal and morphological characterization of the composites was done in place of developing an optimization model due to the following reasons:

1. For this project, 6 weight fractions of lime sludge filler (5%, 10%, 15%, 20%, 25% and 30%) in HDPE composites were considered which was only possible with the given time frame

and amount of funds (1<sup>st</sup> installment from SERB). However, 6 data points are not sufficient as inputs to formulate a robust optimization model capable of correct predictions. Hence, thermal and morphological characterization was done in place of modeling, which provided additional insight towards unveiling various behavioral trends shown by the composites at higher temperatures and under stress.

2. Kindly be informed that the experimental set up was received almost a year after the commencement of the project due to vendor's delay in the delivery of the equipment required for fabrication of composites (injection molding machine and twin screw extruder). Additionally, the mechanical testing of the composites was done in another location (due to absence of testing equipment in-house) which delayed the process further and was also expensive.
3. Moreover, the PI did not receive the second installment of the project (an amount of Rs. 3,55,000.00) which hindered the timely completion of project work (making composites at various other weight fractions of lime sludge for formulating the the model). It may be also be stated that the unspent amount of fund at the end of the project in this case, belonged to the 'Travel expense' category which could not be diverted to other any other heads (required for experimental or computational works).
4. The undersigned had earlier sent letters and emails to SERB (along with the annual progress reports) requesting for an extension of the project time frame. However, no response from SERB was received till date regarding the extension of the time frame.

Hence, the deviation from the proposed objective regarding the formulation of the optimization model was taken.

#### **9. Experimental work giving full details of experimental set up, methods adopted, data collected supported by necessary table, charts, diagrams & photographs:**

The major contribution of this research lies in the fabrication of lime sludge filled polyethylene composite - a novel idea which would focus on integrating the advantages of (i) utilization of paper industry lime sludge waste increasing its commercial viability, (ii) development of a novel polymeric composite; and (iii) decreasing the manufacturing cost of the composite. This research is aimed at exploring the potential of an industrial waste to be used in an alternative application, i.e., as a filler in polymeric matrix. Hence, the mechanical thermal and morphological properties are studied to understand the consequences of adding lime sludge to a polymeric matrix (HDPE). The scientific value added is the development of a new polymeric composite and finding an alternative use of lime sludge waste. Commercial advantage of the work would be to produce polymeric composites of lower cost and to increase the viability of an industrial waste.

This research studies the mechanical, thermal and morphological properties of lime sludge filled HDPE composites in comparison to pure HDPE. The composites are fabricated with increasing weight fraction of lime sludge in a polymeric matrix in order to study the effects of lime sludge on the mechanical, thermal and morphological properties of the composite. It is observed that mechanical properties such as tensile modulus, flexure modulus, flexural strength; and thermal properties such thermal degradation temperature and stability are enhanced as a result of the addition of lime sludge as a filler in HDPE composites.

#### **Experimental work**

Materials used in the fabrication of lime sludge filled HDPE composites, experimental procedures involved and the instrumentation used in the characterization of the composites are described under this section.

## **Material**

HDPE grade 5108L, supplied by Haldia Petrochemicals Ltd. with a melt flow index of 1.9 g/min (@190 °C, 2.16kg) and density of 0.95 g/cm<sup>3</sup> is used as the polymeric matrix. Raw lime sludge powder is collected from the dump yard of the Hindustan Paper Corporation Limited (HPCL) paper mill at Jagiroad, Assam (India). The lime sludge is ground manually with mortar and pestle for 10 min and then passed through a number 170 sieve. The lime sludge is heated at 110 °C for 2 hours to remove any residual moisture content in a hot air oven (model: 114/14, make: Reico) before blending.

## **Sample preparation**

HDPE in form of granules and lime sludge with various weight percents (5, 10, 15, 20, 25 and 30 wt%) is blended together in a co-rotating twin screw extruder (model: EX100, make: Voltam) thereby fabricating lime sludge - HDPE composites of 5, 10, 15, 20, 25 and 30 wt% lime sludge dispersed in a polymeric matrix. The blended polymeric composites in the form of wires are cooled and cut into pellets. Finally, the tensile, flexure and impact test specimens are molded, by using an injection molding machine (model: IM100, make: Voltam) from the pellets. The injection pressure is fixed at 2.45 N/mm<sup>2</sup> and barrel temperature at 200 °C during molding.

## **Mechanical testing**

The tensile and flexural properties are measured using a universal testing machine (model: H100K-S, make: Hounsfield) of capacity 100 kN. Both tensile and flexural testing is carried out at a crosshead speed of 1 mm/min and using a 250N load cell. Four specimens corresponding to each composition of polymeric composites are tested. The average result of the four specimens is reported for each composite. The notched Izod impact test, for the lime sludge - HDPE composite system is conducted using a digital pendulum impact testing machine (model: P/N0963.000, make: CEAST Spa Italy). Tensile, flexure and impact testing of the composites are conducted according to ASTM D638 (specimen V), ASTM D7264 and ASTM D256 standards respectively.

## **DSC analysis**

A differential scanning calorimeter (model: Q20, make: TA instruments, USA) having a standard aluminium pan is used in obtaining the DSC thermograms. The heating rate and cooling rate is maintained at 10 °C/min and nitrogen is used as a sweeping gas. Samples are heated from -70 °C to 200 °C, held isothermally at 200 °C for 1 minute and subsequently cooled to 30 °C. DSC thermograms are plotted for 10 wt% (10 LS HDPE), 20 wt% (20 LS HDPE) and 30 wt% (30 LS HDPE) lime sludge filled HDPE composites and pure HDPE respectively.

## **TGA analysis**

The thermo-gravimetric analysis (TGA) is done by a Shimadzu, Japan thermogravimetric analyzer, TG50. Subsequently, DTA thermograms are calculated from the DSC thermograms. Samples weighing about 6 mg each are heated from 25 °C to 600 °C in nitrogen at a flow rate of 30 ml/min with a heating rate of 10 °C/min. TGA and DTA thermograms are plotted for the samples 10 LS HDPE, 20 LS HDPE, 30 LS HDPE and pure HDPE respectively.

## **X-ray diffraction**

X-ray diffraction study of lime sludge powder sample is conducted at room temperature (27 °C) using a Miniflex 200 (Miniflex, UK) Rigaku X-ray diffractometer with CuK $\alpha$  radiation  $\lambda$ = 1.5418 Å. The scanning rate used is 5 °min<sup>-1</sup> over a range of  $2\theta = 10^\circ$  to  $70^\circ$  for the above study. Peak positions observed on the XRD pattern are indexed according to JCPDS 5-586 of

the International Center for Diffraction Data to search for the presence of  $\text{CaCO}_3$  in lime sludge in its crystalline structure.

### ICP-OES analysis

The elemental contents in lime sludge are estimated using an inductively coupled plasma optical emission spectrometry (ICP-OES) (model: 2100 DV, make: Optima Bridgeport, USA). Elemental concentrations are determined by using an aqueous solution of acid digested lime sludge. 1 g of powdered lime sludge sample is added to 30 mL concentrated nitric acid and 5 mL of concentrated hydrochloric acid. The vessel is immediately closed after acid addition and then the solution is digested inside a hot air oven at  $100^\circ\text{C}$ . Upon digestion of the lime sludge sample, it is cooled and diluted to 100 mL with distilled water.

### SEM analysis

Morphological studies on the fractured surfaces of the tensile specimens of 10LS HDPE, 20 LS HDPE and 30 LS HDPE composites are done using a scanning electron microscope (SEM) (model: JSM-6390LV, make: JEOL) under the secondary electron mode. The samples are sputter coated with platinum to prevent electrical charging before being examined under the SEM.

## 10. Detailed analysis of results indicating contributions made towards increasing the state of knowledge in the subject:

### Results and discussion

Particulate composites of lime sludge and HDPE are fabricated with increasing weight fractions of lime sludge. Mechanical, thermal and morphological properties of the lime sludge – HDPE composites are discussed in this section.

### Characterization of lime sludge

Lime sludge collected from Jagiroad Paper Mill, Assam (India) is characterized using XRD, ICP-OES, SEM, and TGA analysis in order to determine the structure, composition, morphology and thermal properties of lime sludge. Fig. 1 shows the SEM micrograph of lime sludge powder after compounding, which depicts the particle size of lime sludge powder. It is observed from the micrograph that the average size of a lime sludge powder particle is approximately  $2\ \mu\text{m}$ .

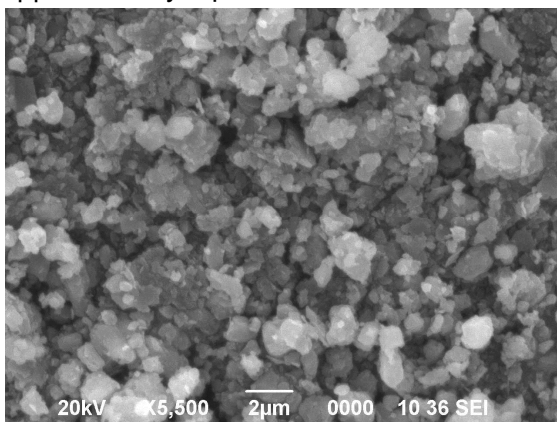


Fig. 1: SEM micrograph of lime sludge powder depicting the particle size

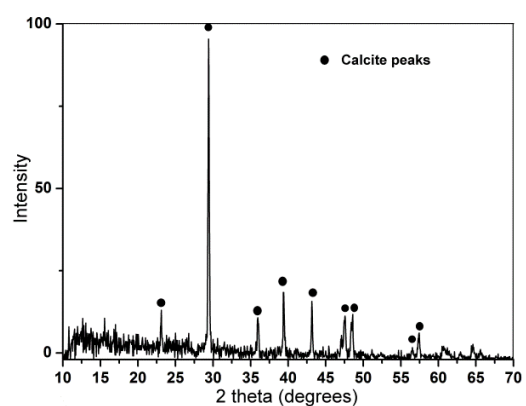


Fig. 2: XRD data of lime sludge showing calcite peaks

### XRD analysis:

The crystal structure of the lime sludge powder is observed by analyzing the results of X-ray

diffraction data (XRD) as shown in Fig. 2. It is observed that the main diffraction peaks of the crystalline phases present in lime sludge are in the range of 20–60°. The experimental XRD data of lime sludge obtained in the present study showed striking resemblance with the diffraction peaks of calcite obtained from JCPDS card 5-586. The most prominent peak of calcite observed in the XRD pattern is located at 29.4° corresponding to the (1 0 4) reflections from the calcite crystals structure. Additionally, calcite peaks are observed at 23.05°, 36°, 39.4°, 43.15°, 47.5°, 48.55°, 56.6° and 57.45° as shown in Fig. 2. These peaks conformed well to the diffraction data of calcite in terms of locations of peaks and their relative intensities. Hence, the observed diffraction data showed the presence of CaCO<sub>3</sub> in lime sludge in the form of calcite. However, the presence of certain other peaks in different locations of the diffraction pattern is due to the fact that lime sludge contains other inorganic compounds in small quantities. Therefore, XRD validates that calcium carbonate in lime sludge is mainly present in the form of calcite crystals, and this makes lime sludge an ideal material to be used as filler in polymer matrices.

**ICP-OES Analysis:**

Elemental analysis done by ICP-OES is shown in Table 1. Elemental analysis showed calcium concentration of 514.5 ppm which is in close conformation with that obtained by Deka and Yasmin [27] in an earlier study on lime sludge obtained from same Jagiroad Paper Mill, Assam (India). Additionally, Deka and Yasmin [1] also reported the percentage of CaCO<sub>3</sub> in the lime sludge to be 66.5% with a water holding capacity of 70.43% along with a few other trace elements. From this analysis it is evident that lime sludge mainly consists of CaCO<sub>3</sub> as the main constituent with traces of a few other compounds.

Table 1: ICP-OES analysis of lime sludge showing elemental concentrations in ppm.

| S. No. | Element | Concentration (ppm) |
|--------|---------|---------------------|
| 1      | Ca      | 514.5               |
| 2      | Fe      | 28.8                |
| 3      | Al      | 51.75               |
| 4      | Mg      | 285.21              |

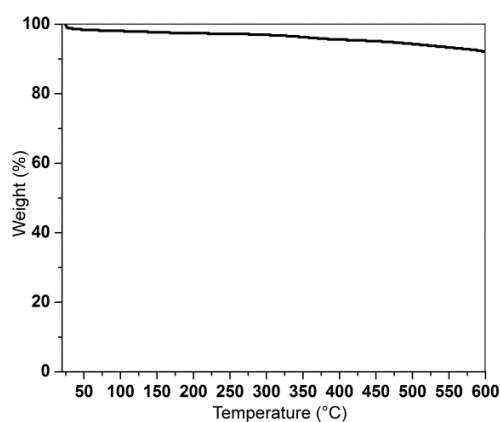


Fig. 3: TGA curve showing the degradation process of lime sludge up to 600°C

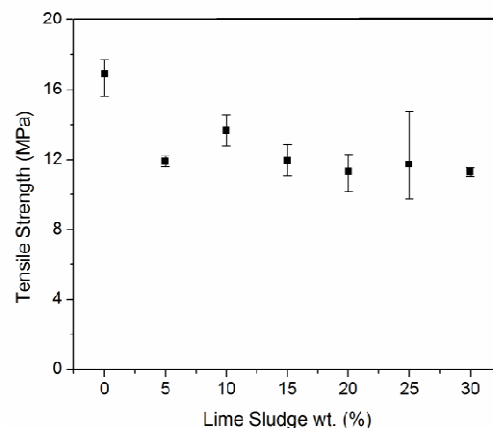


Fig. 4: Variation of tensile strength with increasing lime sludge wt %

**TGA Analysis:**

Thermal degradation of lime sludge is studied using TGA up to a temperature of 600°C. The weight loss % of lime sludge with increase in the temperature is shown in Fig. 3. The TGA analysis is done up to 600°C since thermal degradation of HDPE composites filled with lime sludge are also studied till 600°C and also to assess the thermal stability of lime sludge. It is observed that there is hardly any loss in the weight % of the lime sludge sample up to 600°C. A small loss of 7.82% weight is detected which may be attributed to loss of strongly physically adsorbed water between 40°C to 110°C and loss of water associated with magnesium hydroxide between 200°C and 400°C [2]. Hence, the TGA plot of lime sludge with only a small weight loss of 7.82% indicates the absence of any organic matter (cellulosic fibres) in the lime sludge. It is also provided in the technical report by Baker et al. [2] that upon further heating, the loss between 650°C and 800°C is due to carbon dioxide being driven off as calcium carbonate is decomposed.

**Mechanical properties of the composites**

Tensile, flexure and Izod impact tests are conducted for pure HDPE and lime sludge filled HDPE composites (5, 10, 15, 20, 25 and 30 wt% lime sludge in polymer matrix) in order to determine the mechanical properties of the newly fabricated composites with increasing lime sludge additive in conjunction with the pure polymer. Mechanical properties such as tensile strength, elastic modulus, elongation at break, flexural strength, flexural modulus and Izod impact toughness are measured for the specimens. The results observed were compared with previously reported studies on the mechanical properties of pure CaCO<sub>3</sub> filled polymeric composites.

**Tensile strength:**

The variation in tensile strength with increasing filler loading is shown in Fig. 4. It is found that the tensile strength of pure HDPE is higher than that obtained from all the lime sludge – HDPE polymeric composite. There is a moderate drop in the tensile strength with increasing lime sludge filler loading. This is in conformation with the results obtained by Atikler et al. [3] for pure CaCO<sub>3</sub> filled HDPE composites wherein it was observed that the tensile strength decreased with increasing filler loading. This may be due to dewetting along the filler – matrix boundary may occur due to poor bonding between them. Another probable cause may be larger particle size of CaCO<sub>3</sub> based lime sludge may cause sites of higher stress concentrations and subsequent void creation due to frictional pull of particles, thereby decreasing the tensile strength. Additionally, crazing in polymers is one of the important reasons for decrease in strength and subsequent fracture in polymers under tensile loading [4]. CaCO<sub>3</sub> is known to promote craze formation in deformed polymers before fracture [5]. In this case, lime sludge may also promote crazing in the polymeric composites similar to CaCO<sub>3</sub> which may be due to the presence of CaCO<sub>3</sub> in lime sludge. However, an increase of about 9% in the tensile strength was observed as the filler loading increased from 5 to 10 wt% (11.91 MPa to 13.67 MPa) after which the tensile strength decreases again up to 30% filler loading. This shows that the filler proves to be a better reinforcement as its content increases up to 10% in this case, indicating better particle dispersion and filler-matrix bonding compared to those above 10 wt% filler content.

**Tensile modulus:**

A significant increment in the elastic modulus of composites with increasing filler content is observed, as compared to the lower modulus of pure HDPE. The variation in tensile modulus

with increasing filler loading is shown in Fig. 5. There is a rise in the elastic modulus from 139.6 MPa for pure polymer to 215.2 MPa for 30 wt% lime sludge filled HDPE composite. This increasing trend is similar to that obtained by Atikler et al. [3] which revealed that Young's modulus for 30 wt% pure CaCO<sub>3</sub> filled HDPE increased significantly when compared that that of HDPE alone. In the present case, the increase in Young's modulus for 30 wt% lime sludge filled HDPE composite is about 1.5 times the value for pure HDPE; thus indicating that lime sludge particles provide more rigidity to the composites than pure CaCO<sub>3</sub>. Since the modulus is measured before any significant plastic deformation took place, hence it is independent of the filler – matrix interactions, thereby making the effects of particulate shape and specific surface area immaterial in the measurement of elastic modulus. The rise in the elastic modulus is largely attributed due to the following reasons – (a) replacement of the fully polymeric HDPE matrix with parts of rigid particulate fillers, and (b) introduction of a mechanical restraint due to the restriction in deformation provided by the addition of particulate filler.

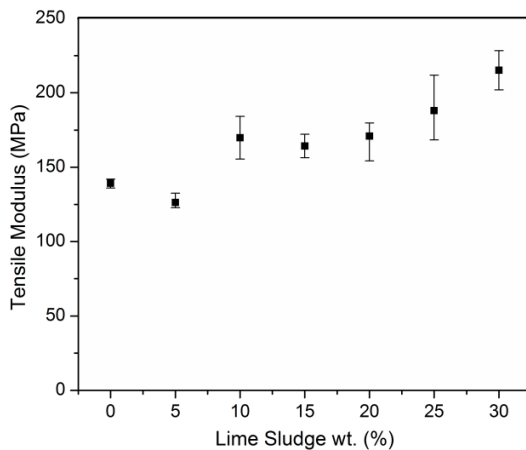


Fig. 5: Variation of tensile modulus with increasing lime sludge wt %

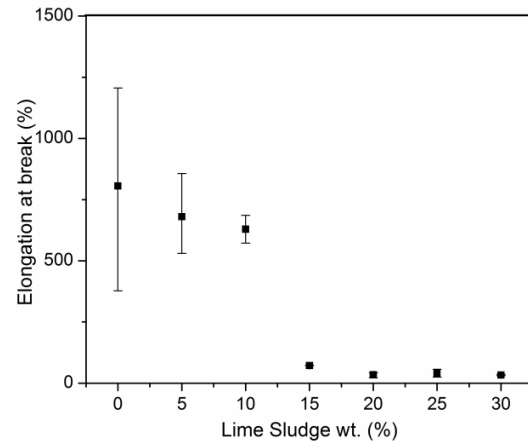


Fig. 6: Variation of elongation at break with increasing lime sludge wt %

#### **Elongation at break:**

It is observed that the elongation at break decreased drastically with increasing filler content which is characteristic of particulate reinforced thermoplastic composites. The plot of elongation at break varying with increasing filler loading is shown in Fig. 6. The decrement in the elongation at break (806% for pure polymer to 32.8% for 30 wt% lime sludge filled HDPE composite) is due to the low elongation of filler that restricts the flow of polymer molecules past one another. This indicates that the mode of failure changes from ductile to brittle behaviour.

Rigidity of particulate fillers, filler agglomeration at 30 wt% lime sludge filled composite, and deformation at selected stress concentration sites at filler-matrix boundary may be attributed to lower elongation at break. Atikler et al. [3] also obtained a similar trend in the elongation % for CaCO<sub>3</sub> filled HDPE composite, wherein the elongation at break % decreased from a value greater than 1000% for pure HDPE to that less than 100% for 30 wt% CaCO<sub>3</sub> filled HDPE composite.

#### **Flexural strength:**

The plot of flexural strength with increasing filler loading is shown in Fig. 7. Increasing order of flexure strength with increasing filler loading (6.23 MPa for pure polymer to 12.19 MPa for 30 wt% lime sludge filled HDPE composite) confirms that lime sludge acts as a better



reinforcement under flexure loading conditions. This indicates better stress transfer between the matrix and filler particles under bending stresses. The increment in flexure strength also indicated better surface properties for lime sludge filled composites since the flexural properties are sensitive to the surface of specimens. This is in conformation with the observed literature that the flexural properties of composites increase with increasing loading of rigid particulate fillers in a polymeric matrix [6].

**Flexural modulus:**

As shown in Fig. 8, incorporation of rigid particulate filler such as lime sludge improved the stiffness of the polymeric composites which resulted in an almost linear increment of flexural modulus (245.7 MPa for pure polymer to 567.5 MPa for 30 wt% lime sludge filled HDPE composite) similar to the tensile modulus. Higher concentration of rigid particulate materials demands higher stress for the same amount of deformation and hence the increase in the flexural modulus. Additionally, flexural modulus of composites is found to be lower than its tensile counterparts, the reason being the presence of excess polymer on the surface as a result of the restriction imposed by the mold walls during molding of the composites. Since the surface of specimens is at maximum stress during flexural analysis, the properties of the surfaces are emphasized more in this case; unlike tensile testing, where the bulk properties of the specimen in the tensile direction are exposed.

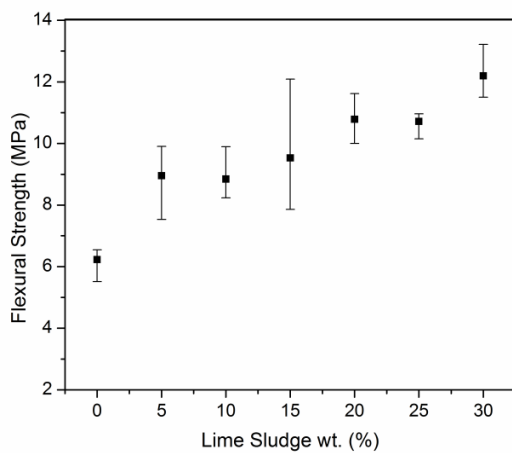


Fig. 7: Variation of flexural strength with increasing lime sludge wt %

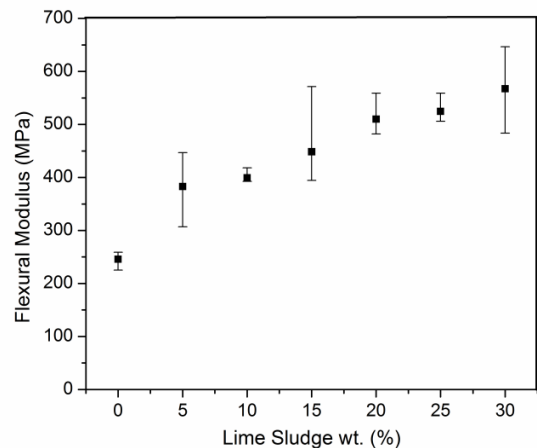


Fig. 8: Variation of flexural modulus with increasing lime sludge wt %

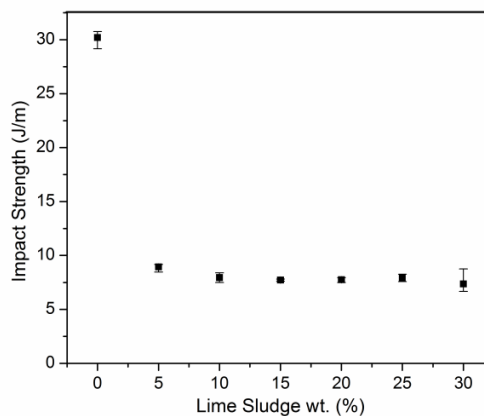


Fig. 9 Variation of Izod impact strength with increasing lime sludge wt %

**Izod impact strength:**

Parameters such as toughness properties of reinforcement, the nature of the interfacial region and the matrix fracture highly influence the impact strength of a composite [7]. As shown in Fig. 9, it is observed that the impact properties decreased with the introduction of filler materials as compared to the pure HDPE samples. It is a well-known fact that the highest stiffness composites (high modulus of elasticity) exhibit the lowest impact resistance since high stress is transferred from the polymeric matrix to the filler particles [8, 9]. The particulate fillers act as points of stress concentration and anisotropic particle orientation throughout the composite restricts plastic deformation, thereby causing embrittlement of the composite. Upon impact, the crack propagates to the interfacial regions and the material fails in a manner brittle as it cannot plastically deform. However, a very low variation of impact strength was observed with increasing filler weight fractions.

It is observed that industrial lime sludge waste used as filler in HDPE provides comparable mechanical properties to pure CaCO<sub>3</sub> filled HDPE composites. Additionally, the rigid lime sludge particles provide better rigidity within the elastic limit than pure CaCO<sub>3</sub> thus increasing the Young's modulus even more. Increment in tensile modulus, flexural strength and modulus justifies lime sludge as not only filler but also as a reinforcing agent in HDPE composites. Hence, industrial lime sludge waste may be used in a polymeric matrix which would increase the viability of an industrial waste and decrease the cost of the composites without much compromise in the mechanical properties.

**Thermal properties of the composites**

Thermal properties of lime sludge – HDPE composites are studied using DSC and TGA techniques. The thermal properties of three composites (10 LS HDPE, 20 LS HDPE and 30 LS HDPE) are studied in comparison with pure HDPE samples.

**DSC analysis:**

DSC measurements are conducted in order to determine the thermal properties such as melting temperature ( $T_m$ ), onset melting temperature ( $T_{on}$ ), heat of fusion ( $H_m$ ), heat of crystallization ( $H_c$ ), degree of crystallinity, onset crystallization temperature ( $T_{con}$ ), and peak crystallization temperature ( $T_c$ ). Comparative studies on DSC thermograms of pure HDPE, 10% (10 LS HDPE), 20% (20 LS HDPE) and 30% (30 LS HDPE) lime sludge filled HDPE composites are carried out. From the recorded heating and cooling curves for these composites as shown in Figs. 10(a) and 10(b) respectively, the thermal properties are calculated and tabulated as shown in Table 2. The percentage of crystallinity ( $\chi_c$ ) in pure HDPE and its composites are calculated based on the formula provided by Liu et al. [10] as shown in Eq. (1).

$$\chi_c = \frac{H_m}{(H_m^\circ \times w)} \times 100 \quad (1)$$

where  $H_m^\circ$  is the heat of fusion for 100% crystalline HDPE, i.e., 293 J/g [11] and  $w$  is the mass fraction of HDPE in the composites.

It is observed that there is a slight increment in the melting temperatures and the onset melting temperatures of the composites with the addition of the lime sludge filler in the composites. The onset melting temperature increased from 121.72°C to 123.88°C and the melting temperature increased from 126.83°C to 132.17°C with the addition of filler. The melting enthalpy decreased with the increasing loading of filler content indicating the required heat to melt the polymeric composite decreased. This translates into money and power

savings during extrusion and molding of polymeric composite fabrication [12]. It is also observed that the crystallization peak temperatures also increased with the increasing addition of lime sludge filler (from 113.30°C to 115.32°C) indicating that lime sludge could act as a nucleating agent, thus increasing the crystallization temperature of HDPE. It is a well-known fact that one of the significant effects of particulate fillers on the crystallinity of semi-crystalline polymers is their capability to act as nucleating agents [6, 13-14]. However, calculation of the percentage crystallinity of pure HDPE and each of the composites (10%, 20% and 30% filled lime sludge) reveals that percentage crystallinity decreased with the addition of increasing filler loading. Hence, it is evident that in the present case, lime sludge particles reduce the conformational changes available to the polymer macromolecules during crystallization. CaCO<sub>3</sub> present in lime sludge restricts the macromolecular ability thereby, reducing the spaces available for the polymeric macromolecules to arrange themselves according to statistical thermodynamics [12, 15].

Lime sludge induced restriction in the movement of polymer chain segments and depletion of the free spaces available for the macromolecules to occupy them, retards crystal growth [42]. This reduction in the conformational changes can be attributed to the decrease in the entropy of crystallization ( $\Delta S_c$ ) given in Equation 2.

$$\Delta S_c = \frac{H_c}{T_c} \quad (2)$$

where,  $H_c$  is the crystallization enthalpy and  $T_c$  is the peak crystallization temperature. As seen in Table 2, it is evident that  $\Delta S_c$  decreased with increasing lime sludge filler loading which may be attributed to the high content of CaCO<sub>3</sub> present in the lime sludge which reduces the free volume/freedom available to the macromolecules (retarding crystal growth) [16-18]. The decrease in crystallinity index also indicates lesser heat required for melting during extrusion and molding, thus contributing to power saving and financial benefits. Herein, values of  $\Delta S_c$  denote the reduction in the degree of freedom available for the movement of macromolecules contributing to crystal growth. From the thermal analysis, it is evident that there are two contrasting properties exhibited by the polymeric composites due to the addition of CaCO<sub>3</sub> rich lime sludge - (a) acting as nucleating agent and (b) crystal growth retarders [16]. In this case, it is evident that the effect of lime sludge as a crystal growth retarder far surpasses its ability to act as nucleating agent. Hence, a decrease in the degree of crystallinity with increasing filler loading is observed.

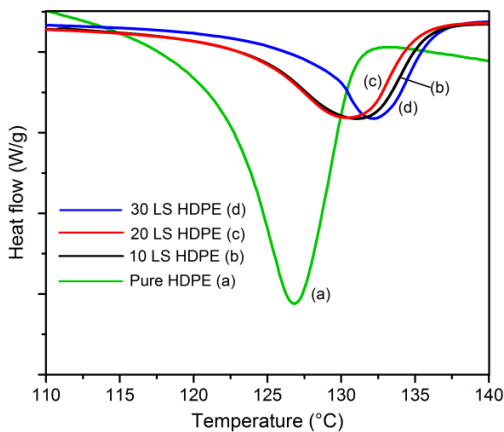


Fig. 10(a): DSC thermogram during heating cycle for composites and pure HDPE

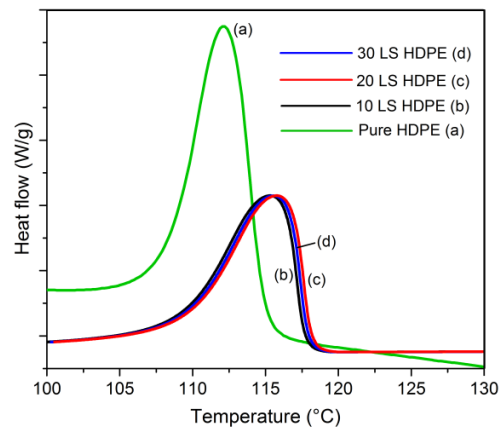


Fig. 10(b): DSC thermogram during cooling cycle for composites and pure HDPE

Table 2: DSC results (heating and cooling) of pure HDPE and lime sludge – HDPE composites

|            | Heating       |            |                |              | Cooling        |             |            | $\Delta S_c$<br>(J/g°C) |
|------------|---------------|------------|----------------|--------------|----------------|-------------|------------|-------------------------|
|            | $T_{on}$ (°C) | $T_m$ (°C) | $H_m$<br>(J/g) | $\chi_c$ (%) | $T_{con}$ (°C) | $H_c$ (J/g) | $T_c$ (°C) |                         |
| Pure HDPE  | 121.72        | 126.83     | 228.10         | 77.66        | 115.51         | 205.93      | 112.10     | 1.84                    |
| 10LS HDPE  | 120.92        | 131.40     | 152.64         | 57.77        | 116.95         | 151.30      | 114.75     | 1.32                    |
| 20 LS HDPE | 121.08        | 130.04     | 124.38         | 52.94        | 117.61         | 133.60      | 114.98     | 1.16                    |
| 30 LS HDPE | 123.88        | 132.17     | 107.04         | 52.08        | 117.70         | 121.80      | 115.32     | 1.05                    |

### TGA analysis:

Thermal parameters such as thermal degradation, onset temperature, temperature at the maximum rate of weight loss and % residual weight left at 600 °C are used in order to conduct comparative analysis of thermal decomposition between lime sludge - HDPE composites and pure HDPE.

TGA analysis is conducted in order to study the thermal stability of lime sludge filled HDPE composites in comparison with pure HDPE. TGA (weight% vs temperature) and DTG (Derivative of weight% vs temperature) thermograms are plotted as shown in Figures 11(a) and 11(b) respectively. The results calculated from the thermograms for each composition of composites and pure HDPE are tabulated as shown in Table 3. From the thermograms it is observed that the thermal stability of the composites increased with increasing weight percentage of lime sludge in the HDPE matrix. This is evident from the fact that the onset temperature for thermal degradation ( $T_{on}$ ) increased from 429.38 °C for pure HDPE to 448.28 °C for 30% lime sludge filled HDPE composites. Additionally, the temperature at which maximum weight loss % ( $T_{max}$ ) also increased with increasing lime sludge percentage in the composites indicating that the initial degradation product is the polymer, while further degradation in the composites are absorbed by the lime sludge, thus increasing the temperature of the point of maximum degradation. This is also confirmed by the amount of residual weight % of material left at 600 °C which increased from 0.69% for pure HDPE to 21.6% for 30 LS HDPE.

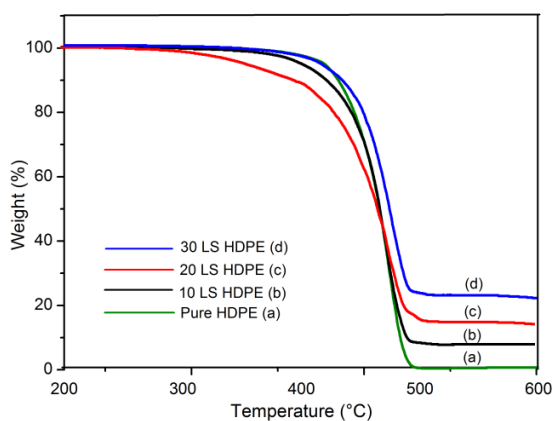


Fig. 11(a): TGA plot for lime sludge – HDPE composites and pure HDPE

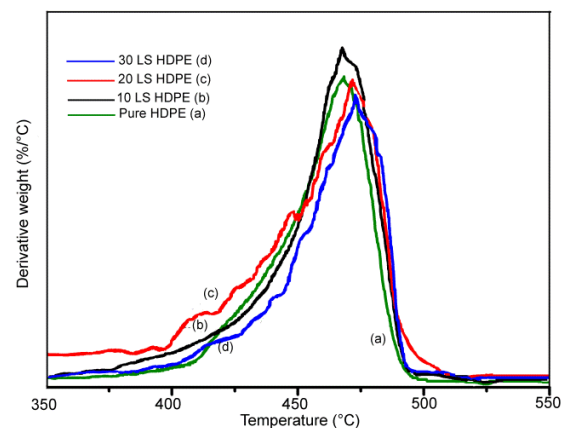


Fig. 11 (b): DTG plot for lime sludge – HDPE composites and pure HDPE

Table 3: TGA results of pure HDPE and lime sludge – HDPE composites

| Material   | Residual weight % | T <sub>max</sub> (°C) | T <sub>on</sub> (°C) |
|------------|-------------------|-----------------------|----------------------|
| Pure HDPE  | 0.69              | 468.27                | 429.38               |
| 10 LS HDPE | 7.75              | 468.92                | 430.91               |
| 20 LS HDPE | 14.50             | 471.90                | 434.94               |
| 30 LS HDPE | 21.60             | 476.33                | 448.28               |

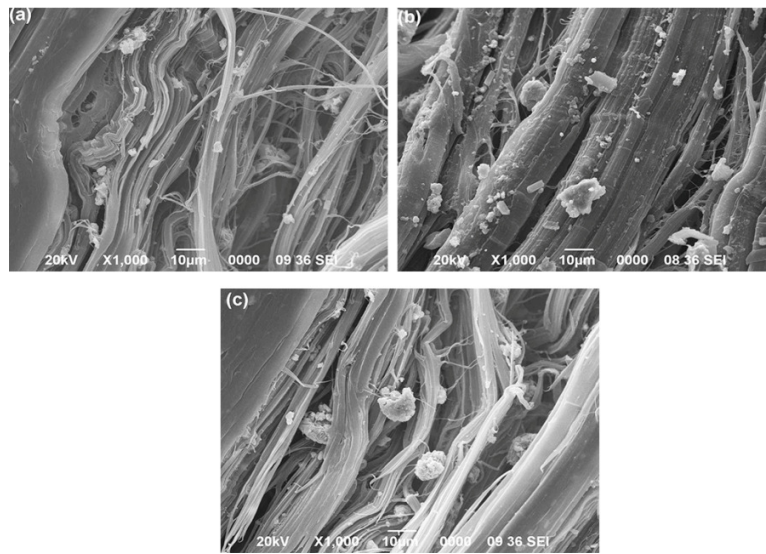


Fig. 12: SEM micrograph at the fractured portion of the tensile test specimen for (a) 10 LS HDPE, (b) 20 LS HDPE, (c) 30 LS HDPE composites

### Morphological analysis of the composites

Effective dispersion of the filler in a polymeric matrix and bonding at the filler – matrix interface are two prime parameters affecting the mechanical properties of polymeric composites. As observed from the micrograph of lime sludge particles in Fig. 1, the average size of a lime sludge powder particle is approximately 2 µm. Figs. 12(a), 12(b) and 12(c) show the secondary electron micrographs of the three composites - 10 LS HDPE, 20 LS HDPE and 30 LS HDPE respectively, obtained at the elongated fractured portion of the respective tensile test specimens. It is observed that with increasing amount of lime sludge in the composite, the tendency of the filler particles to agglomerate also increased. The particle dispersion and size is uniform in 10 LS HDPE and 20 LS HDPE composites when compared with 30 LS HDPE (Fig. 12(c)) where particle agglomeration is observed. Fig. 12(c) shows that for 30 wt% lime sludge filled HDPE composite, the lime sludge particle size is approximately 10 µm which indicates agglomeration of particles. This is in conformation with the fact that the tensile strength decreased for 30 LS HDPE composite due to poor particle dispersion and higher filler particle size which increased the stress concentrations at localized regions causing embrittlement of the composite. Dewetting due to debonding of the filler and matrix at the interface is clearly evident from the micrographs. It is also observed that lime sludge particles fell out of the matrix at isolated locations creating voids. This condition of poor adhesion between the filler and matrix is in correlation with the fact that the tensile strength of pure

polymer was higher than that of the composites as debonding occurred due to the non-adherence of filler particles to the matrix. Thus, the filler particles are unable to handle the stress upon loading leading to lower tensile strength observed in polymeric composites.

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4. Misra R, Nerikar P, Bertrand K, Murphy D (2004) Some aspects of surface deformation and fracture of 5–20% calcium carbonate reinforced polyethylene composites. *Mater Sci Eng A - Struct* 384(1):284–298.
5. Leong Y, Ishak Z, Ariffin A (2004) Mechanical and thermal properties of talc and calcium carbonate filled polypropylene hybrid composites. *J Appl Polym Sci* 91(5):3327–3336.
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8. Riley A, Paynter C, McGenity P, Adams JM (1990) Factors affecting the impact properties of mineral filled polypropylene. *Plast Rub Proc Appl* 14(2):85–93.
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## 11. Conclusions summarizing the achievements and indication of scope for future work:

This study revealed that lime sludge industrial waste is an attractive option to be used as reinforcing filler in polymeric composites. This will enhance the functionality and commercial viability of both the filler and the polymer by - (a) usage of lime sludge waste as filler, and (b) decreasing the manufacturing cost of polymer composites. Low particle size, higher thermal stability and mechanical rigidity provided by lime sludge in a polymeric composite in addition to its wide scale availability makes it an ideal filler to produce elastically rigid composites with superior thermal properties and flexural properties at a reduced cost. Results revealed that addition of lime sludge in HDPE matrix significantly increased the tensile and flexural modulus which can be attributed to increased stiffness due to the addition of rigid particulate fillers providing mechanical restraint to deformation. Additionally, the flexural strength also increased indicating better stress transfer between filler and matrix under bending stresses. Thermal stability of the composites increased with the addition lime sludge filler which can be attributed to the increase in onset temperature of thermal degradation, melting temperature and residual weight% with the increase in filler content in the composites. Thus, use of lime sludge filler in HDPE matrix can enhance various mechanical and thermal properties, in addition to decreasing the cost of polymeric composite; reduce pollution and increasing the functionality of an industrial waste.

## 12. S&T benefits accrued:

### i. List of Research publications

| S No | Authors                   | Title of paper  | Name of the Journal                              | Volume                   | Pages | Year |
|------|---------------------------|---|--|--------------------------|-------|------|
| 1.   | Kashyap, S. and Datta, D. | Injection molding process optimization: a review  | The International Journal of Plastics Technology | 18                       | 1-18  | 2015 |
| 2.   | Kashyap, S. and Datta, D. | Reusing industrial lime sludge waste as a filler in polymeric composites                              | Materials Today: Proceedings                     | Accepted for publication |       |      |
| 3.   | Kashyap, S. and Datta, D. | Lime sludge waste-HDPE composites - a study of their mechanical, thermal and morphological properties | Submitted to Journal (Under review)              |                          |       |      |

- ii. Manpower trained on the project
- a) Research Scientists or Research Associates: Nil
- b) No. of Ph.D. produced: Nil
- c) Other Technical Personnel trained: Nil
- iii. Patents taken, if any Nil

### 13. Financial Position:

Kindly note that an amount of Rs. 2,13,780.00 which was left unspent from the capital (equipment) head of Rs. 18,85,000.00 was divided into the categories of Consumables (Rs. 81,250.00), Contingency (Rs. 39,000.00) and Overhead expenses (Rs. 93,530.00). This was done with permission from SERB (Dr. Prabhakar Mohanty). The permission received via e-mail to this request is also enclosed herewith.

| No. | Financial Position/ Budget Head (Rs.) | Funds Sanctioned (Rs.) | Funds Received from DST-SERB (Rs.)   | Expenditure (Rs.)   | % of Total cost |
|-----|---------------------------------------|------------------------|--|---------------------|-----------------|
| I   | Salaries/ Manpower costs              | Nil                    | Nil  | Nil                 | 0               |
| II  | Equipment                             | 18,85,000.00           | 16,71,220.00<br>(extra amount of Rs. 2,13,780.00* transferred to other sections) | 16,71,220.00        | 80.54           |
| III | Supplies & Materials                  | 1,25,000.00            | 43,750.00 + 81,250.00* = 1,25,000.00   | 1,24,996.00         | 6.02            |
| IV  | Contingencies                         | 60,000.00              | 21,000.00 + 39,000.00* = 60,000.00   | 62,216.00**         | 2.99            |
| V   | Travel                                | 60,000.00              | 21,000.00  | 9,788.00            | 0.47            |
| VI  | Overhead Expenses                     | 3,00,000.00            | 1,04,250.00 + 93,530.00* = 1,97,780.00   | 1,95,529.00         | 9.42            |
| VII | Others, if any                        | Nil                    | Nil  | Nil                 | 0               |
|     | <b>Total</b>                          | <b>24,30,000.00</b>    | <b>20,75,000.00</b>  | <b>20,63,749.00</b> | <b>99.44%</b>   |

**NOTE:**

\* transferred from the Equipment head (unspent amount of Rs. 2,13,780.00) to Consumables (Rs. 81,250.00), Contingency (Rs. 39,000.00) and Overhead expenses (Rs. 93,530.00) with permission from SERB.

\*\* An extra amount Rs. 2,216.00 spent from "Contingency" is adjusted from the "Overhead" section.



## 14. Procurement/ Usage of Equipment

a)

| S. No. | Name of Equipment                        | Procured (Yes/No)<br>Model & Make                            | Cost (in lakhs) | Working (Yes/No) | Utilisation rate (%) | Remarks regarding maintenance/breakdown |
|--------|--|--|-----------------|------------------|----------------------|---|
| 1.     | Injection Molding Machine                | Yes,<br>M/s Voltam Furnace Industries, Kolkata               | 10.069          | Yes              | 100%                 | Working satisfactorily                  |
| 2.     | Twin Screw Extruder                      |  |                 | Yes              | 100%                 | Require maintenance                     |
| 3.     | Rectangular Muffle furnace               | Yes,<br>M/s Reico Equipment and Instrument Pvt. Ltd, Kolkata | 0.435           | Yes              | 90%                  | Working satisfactorily                  |
| 4.     | Hot Air Oven                             |  |                 | Yes              | 90%                  | Working satisfactorily                  |
| 5.     | Optical Microscope with imaging software | Yes,<br>VISION metallurgical microscope<br>Model: VINV-MET   | 5.1296          | Yes              | 50%                  | Working satisfactorily                  |
| 6.     | Laptop                                   | Yes,<br>HP Probook 4440S with Core i5 Processor              | 0.63            | Yes              | 100%                 | Working satisfactorily                  |
| 7.     | Printer                                  | HP Colour Laserjet Pro 451DN                                 | 0.4485          | Yes              | 100%                 | Working satisfactorily                  |

### Photos of major Equipment purchased:



Fig.: Twin screw extruder



Fig.: Laboratory injection molding machine



Fig.: Muffle furnace



Fig.: Hot air oven

### **b) Plans for utilizing the equipment facilities in future**

All the equipment procured for the project will be used in the department for teaching and research work of B.Tech and M.Tech students as a part of their curriculum. The equipment will also be used for demonstrations in the fabrication of composites in courses related to composite materials. Moreover, the equipment facilities will also aid the research work of various faculty and students in Tezpur University in the long run.

*Satadru Kashyap*

Satadru Kashyap dated 04.12.16


(Principal Investigator)

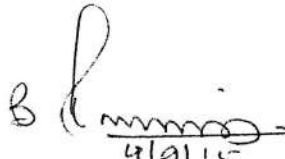
**UTILISATION CERTIFICATE**  
**(TO BE SENT IN DUPLICATE TO DEPARTMENT OF SCIENCE AND TECHNOLOGY)**  
**For the financial year 2013 -2014**


|                      |  |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
|----------------------|--|--|----------------------|--------------|--------------|-------------|----------------|-----------|---------|-----------|-----------|-------------|----------------|------------------------|
| 1.                   | Title of the Project/Scheme  | Utilization of the Calcium Carbonate obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 2.                   | Name of the institution  | TEZPUR UNIVERSITY  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 3.                   | Principal Investigator   | Satadru Kashyap  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 4.                   | Dept. of Science and Technology sanction order no. and date of sanctioning of the project  | SB/FTP/ETA-88/2013 dated 03.09.2013  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 5.                   | Head of the account as given in the original sanction order  | <table style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 70%;">Capital (Equipment):</td> <td style="text-align: right;">18,85,000.00</td> </tr> <tr> <td>Consumables:</td> <td style="text-align: right;">1,25,000.00</td> </tr> <tr> <td>Contingencies:</td> <td style="text-align: right;">60,000.00</td> </tr> <tr> <td>Travel:</td> <td style="text-align: right;">60,000.00</td> </tr> <tr> <td>Overhead:</td> <td style="text-align: right;">3,00,000.00</td> </tr> <tr> <td><b>Total :</b></td> <td style="text-align: right;"><b>Rs 24,30,000.00</b></td> </tr> </table> | Capital (Equipment): | 18,85,000.00 | Consumables: | 1,25,000.00 | Contingencies: | 60,000.00 | Travel: | 60,000.00 | Overhead: | 3,00,000.00 | <b>Total :</b> | <b>Rs 24,30,000.00</b> |
| Capital (Equipment): | 18,85,000.00   |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| Consumables:         | 1,25,000.00  |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| Contingencies:       | 60,000.00  |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| Travel:              | 60,000.00  |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| Overhead:            | 3,00,000.00  |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| <b>Total :</b>       | <b>Rs 24,30,000.00</b>   |  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 6.                   | Amount brought forward from the previous financial year quoting DST letter No. and date in which the authority to carry forward the said amount was given. | Not Applicable   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 7.                   | Amount received during the financial year (Please give DST letter/order no. and date)  | Rs. 20,75,000.00<br>Letter No. SB/FTP/ETA-88/2013 dated 03.09.2013   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 8.                   | Total amount that was available for expenditure (excluding commitments) during the financial year (Sr. no. 6+7)  | Rs. 20,75,000.00   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 9.                   | Actual expenditure (excluding commitments) incurred during the financial year (up to 31 <sup>st</sup> March, 2014)   | Rs. 62,500.00  |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 10.                  | Balance amount available at the end of the financial year  | Rs. 20,12,500.00   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 11.                  | Unspent balance refunded, if any (please give the details of Cheque No. etc)   | Not applicable   |                      |              |              |             |                |           |         |           |           |             |                |                        |
| 12.                  | Amount to be carried forward to the next financial year 2014-15 (if applicable)  | Rs. 20,12,500.00   |                      |              |              |             |                |           |         |           |           |             |                |                        |

## UTILISATION CERTIFICATE

Certified that the amount of Rs. 20,75,000.00 of grant sanctioned during the year **2013 -14** in favor of Registrar, Tezpur University vide sanction order no SB/FTP/ETA-88/2013 on 03.09.2013, a sum of Rupees **62,500.00** (for financial year 2013-2014) has been utilized for the purpose of Research project for which it was sanctioned and that the balance of Rs. 20,12,500.00 remaining unutilized at the end of the year may be permitted to be utilized during the year 2014-15.

  
(SATADRU KASHYAP)  
Name & Signature  
of PI

  
Name & Signature  
of Accounts Officer with seal  
Finance Unit.  
Tezpur University

  
Name & Signature of  
Head of the Institution  
With date and seal  
Registrar  
Tezpur University

### (TO BE FILLED BY DST)

Certified that I have satisfied myself that the conditions on which the grant was sanctioned have been fulfilled/ are being fulfilled and that I have exercised the following checks to see that the money was actually utilized for which it was sanctioned.

Kinds of checks exercised

- i)
- ii)

Signature  
Designation  
Date:

**STATEMENT OF EXPENDITURE**  
2013 -14

| S. No | Sanctioned Heads              | Funds Allocated     | Funds Received      | Expenditure Incurred |  |  |  |  |  | Total Expenditure | Balance as on date  | Requirement of funds upto 31st March, 2016 | Remark (if any) |
|-------|-------------------------------|---------------------|---------------------|----------------------|--|--|--|--|--|-------------------|---------------------|--|-----------------|
|       |                               |                     |                     | 2013-2014            |  |  |  |  |  |                   |                     |  |                 |
| 1     | CAPITAL (Equipment)           | 18,85,000.00        | 18,85,000.00        | Nil                  |  |  |  |  |  | -                 | 18,85,000.00        | -  |                 |
|       | Non-recurring Items (CAPITAL) | 18,85,000.00        | 18,85,000.00        | Nil                  |  |  |  |  |  | -                 | 18,85,000.00        | -  |                 |
| 2     | CONSUMABLES                   | 1,25,000.00         | 43,750.00           | Nil                  |  |  |  |  |  | -                 | 43,750.00           | 81,250.00                                  |                 |
| 3     | CONTINGENCIES                 | 60,000.00           | 21,000.00           | Nil                  |  |  |  |  |  | -                 | 21,000.00           | 39,000.00                                  |                 |
| 4     | TRAVEL                        | 60,000.00           | 21,000.00           | Nil                  |  |  |  |  |  | -                 | 21,000.00           | 39,000.00                                  |                 |
| 5     | OVERHEAD                      | 3,00,000.00         | 1,04,250.00         | 62,500.00            |  |  |  |  |  | -                 | 41,750.00           | 1,95,750.00                                |                 |
|       | Recurring Items (GENERAL)     | 5,45,000.00         | 1,90,000.00         | 62,500.00            |  |  |  |  |  | 62,500.00         | 1,27,500.00         | 3,55,000.00                                |                 |
|       | <b>TOTAL</b>                  | <b>24,30,000.00</b> | <b>20,75,000.00</b> | <b>62,500.00</b>     |  |  |  |  |  | <b>62,500.00</b>  | <b>20,12,500.00</b> | <b>3,55,000.00</b>                         |                 |

NOTE:

*Satish Kumar*  
Signature of PI

*B. P. Kumar*  
Signature of Accounts Officer  
Finance Officer  
Tazara University

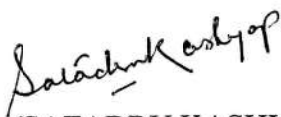
*B. P. Kumar*  
Signature of the Head of the Institute  
Tazara University

**UTILISATION CERTIFICATE**  
**(TO BE SENT IN DUPLICATE TO DEPARTMENT OF SCIENCE AND TECHNOLOGY)**  
**For the financial year 2014 -2015**


|     |  |  |
|-----|--|--|
| 1.  | Title of the Project/Scheme  | Utilization of the Calcium Carbonate obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms |
| 2.  | Name of the institution  | TEZPUR UNIVERSITY  |
| 3.  | Principal Investigator   | Satadru Kashyap  |
| 4.  | Dept. of Science and Technology sanction order no. and date of sanctioning of the project  | SB/FTP/ETA-88/2013 dated 03.09.2013  |
| 5.  | Head of the account as given in the original sanction order  | Capital (Equipment): 18,85,000.00<br>Consumables: 1,25,000.00<br>Contingencies: 60,000.00<br>Travel: 60,000.00<br>Overhead: 3,00,000.00<br><br>Total : Rs 24,30,000.00   |
| 6.  | Amount brought forward from the previous financial year quoting DST letter No. and date in which the authority to carry forward the said amount was given. | Rs. 20,12,500.00   |
| 7.  | Amount received during the financial year (Please give DST letter/order no. and date)  | Not Applicable   |
| 8.  | Total amount that was available for expenditure (excluding commitments) during the financial year (Sr. no. 6+7)  | Rs. 20,12,500.00   |
| 9.  | Actual expenditure (excluding commitments) incurred during the financial year (up to 31 <sup>st</sup> March, 2015)   | Rs. 16,91,358.00   |
| 10. | Balance amount available at the end of the financial year  | Rs. 3,21,142.00  |
| 11. | Unspent balance refunded, if any (please give the details of Cheque No. etc)   | Not applicable   |
| 12. | Amount to be carried forward to the next financial year 2014-15 (if applicable)  | Rs. 3,21,142.00  |

## UTILISATION CERTIFICATE

Certified that the amount of Rs. 20,75,000.00 of grant sanctioned during the year **2013 -14** in favor of Registrar, Tezpur University vide sanction order no SB/FTP/ETA-88/2013 on 03.09.2013, a sum of Rupees 16,91,358.00 (for financial year 2014-2015) has been utilized for the purpose of Research project for which it was sanctioned and that the balance of Rs. 3,21,142.00 remaining unutilized at the end of the year may be permitted to be utilized during the year 2015-16.

  
(SATADRU KASHYAP)  
Name & Signature  
of PI

  
9/9/15  
Name & Signature  
of Accounts Officer with seal  
Finance Officer  
Tezpur University

  
Name & Signature of  
Head of the Institution  
With date and seal  
Registrar  
Tezpur University

**(TO BE FILLED BY DST)**

Certified that I have satisfied myself that the conditions on which the grant was sanctioned have been fulfilled/ are being fulfilled and that I have exercised the following checks to see that the money was actually utilized for which it was sanctioned.

Kinds of checks exercised

- i)
- ii)

Signature  
Designation  
Date:

**STATEMENT OF EXPENDITURE**  
2013 -14 & 2014-15

| S. No | Sanctioned Heads                 | Funds Allocated     | Funds Received      | Expenditure Incurred |   | Total Expenditure   | Balance as on date | Requirement of funds upto 31st March, 2016 | Remark (if any) |
|-------|----------------------------------|---------------------|---------------------|----------------------|---|---------------------|--------------------|--|-----------------|
|       |                                  |                     |                     | 2013-2014            | 2014 -15<br>(till 31 <sup>st</sup> March, 2015) |                     |                    |  |                 |
| 1     | CAPITAL<br>(Equipment)           | 18,85,000.00        | 18,85,000.00        | Nil                  | 16,71,220.00                                    | 16,71,220.00        | 2,13,780.00        | -  |                 |
|       | Non-recurring Items<br>(CAPITAL) | 18,85,000.00        | 18,85,000.00        | Nil                  | 16,71,220.00                                    | 16,71,220.00        | 2,13,780.00        | -  |                 |
| 2     | CONSUMABLES                      | 1,25,000.00         | 43,750.00           | Nil                  | -   | -                   | 43,750.00          | 81,250.00                                  |                 |
| 3     | CONTINGENCIES                    | 60,000.00           | 21,000.00           | Nil                  | 20,138.00                                       | 20,138.00           | 862.00             | 39,000.00                                  |                 |
| 4     | TRAVEL                           | 60,000.00           | 21,000.00           | Nil                  | -   | -                   | 21,000.00          | 39,000.00                                  |                 |
| 5     | OVERHEAD                         | 3,00,000.00         | 1,04,250.00         | 62,500.00            | -   | -                   | 41,750.00          | 1,95,000.00                                |                 |
|       | Recurring Items (GENERAL)        | 5,45,000.00         | 1,90,000.00         | 62,500.00            | 20,138.00                                       | 20,138.00           | 1,07,362.00        | 3,55,000.00                                |                 |
|       | <b>TOTAL</b>                     | <b>24,30,000.00</b> | <b>20,75,000.00</b> | <b>62,500.00</b>     | <b>16,91,358.00</b>                             | <b>17,53,858.00</b> | <b>3,21,142.00</b> | <b>3,55,000.00</b>                         |                 |

NOTE:

*Satish Kumar*  
Signature of PI

*B. K. M. M.*  
Signature of Accounts Officer

Finance Officer  
Tatyasaheb Kore  
Tatyasaheb Kore


*B. K. M. M.*  
Signature of the Head of the Institute

Registrar  
Tatyasaheb Kore  
Tatyasaheb Kore



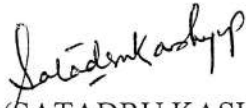
**UTILISATION CERTIFICATE**  
**(TO BE SENT IN DUPLICATE TO DEPARTMENT OF SCIENCE AND TECHNOLOGY)**  
**For the financial year 2015 -2016**

|     |  |  |
|-----|--|--|
| 1.  | Title of the Project/Scheme  | Utilization of the Calcium Carbonate obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms |
| 2.  | Name of the institution  | TEZPUR UNIVERSITY  |
| 3.  | Principal Investigator   | Satadru Kashyap  |
| 4.  | Dept. of Science and Technology sanction order no. and date of sanctioning of the project  | SB/FTP/ETA-88/2013 dated 03.09.2013  |
| 5.  | Head of the account as given in the original sanction order  | Capital (Equipment): 18,85,000.00<br>Consumables: 1,25,000.00<br>Contingencies: 60,000.00<br>Travel: 60,000.00<br>Overhead: 3,00,000.00  |
|     |  | Total : Rs 24,30,000.00  |
| 6.  | Amount brought forward from the previous financial year quoting DST letter No. and date in which the authority to carry forward the said amount was given. | Rs. 3,21,142.00  |
| 7.  | Amount received during the financial year (Please give DST letter/order no. and date)  | Not Applicable   |
| 8.  | Total amount that was available for expenditure (excluding commitments) during the financial year (Sr. no. 6+7)  | Rs. 3,21,142.00  |
| 9.  | Actual expenditure (excluding commitments) incurred during the financial year (up to 31 <sup>st</sup> March, 2016)   | Rs. 2,55,213.00  |
| 10. | Balance amount available at the end of the financial year  | Rs. 65,929.00  |
| 11. | Unspent balance refunded, if any (please give the details of Cheque No. etc)   | Not applicable   |
| 12. | Amount to be carried forward to the next financial year 2016-17 (if applicable)  | Rs. 65,929.00  |


  
 21/6/16  
**Finance Officer**  
 Tezpur University

## UTILISATION CERTIFICATE

Certified that the amount of Rs. 20,75,000.00 of grant sanctioned during the year **2013 -14** in favor of Registrar, Tezpur University vide sanction order no SB/FTP/ETA-88/2013 on 03.09.2013, a sum of Rupees 2,55,213.00 (for financial year 2015-2016) has been utilized for the purpose of Research project for which it was sanctioned and that the balance of Rs. 65,929.00 remaining unutilized at the end of the year may be permitted to be utilized during the year 2016-17.

  
(SATADRU KASHYAP)  
Name & Signature  
of PI

  
Name & Signature  
of Accounts Officer with seal  
Finance Officer  
Tezpur University

  
Name & Signature of  
Head of the Institution  
With date and seal  
Registrar  
Tezpur University

**(TO BE FILLED BY DST)**

Certified that I have satisfied myself that the conditions on which the grant was sanctioned have been fulfilled/ are being fulfilled and that I have exercised the following checks to see that the money was actually utilized for which it was sanctioned.

Kinds of checks exercised

- i)
- ii)


Signature  
Designation  
Date:


**STATEMENT OF EXPENDITURE**  
2013 -14, 2014-15 & 2015-16


| S. No | Sanctioned Heads              | Funds Allocated | Funds Received                             | Expenditure Incurred |  |   | Total Expenditure | Balance as on date | Requirement of funds upto September, 2016-17 | Remark (if any)  |
|-------|-------------------------------|-----------------|--|----------------------|--|---|-------------------|--------------------|--|--|
|       |                               |                 |  | 2013-2014            | 2014 -15 (till 31 <sup>st</sup> March, 2015) | 2015-16 (till 31 <sup>st</sup> March, 2015) |                   |                    |  |  |
| 1     | CAPITAL (Equipment)           | 18,85,000.00    | 16,71,220.00 + 2,13,780.00* = 18,85,000.00 | Nil                  | 16,71,220.00                                 | Nil   | 16,71,220.00      | Nil                | -  | Balanced of 2,13,780.00 transferred to Recurring heads |
|       | Non-recurring Items (CAPITAL) | 18,85,000.00    | 16,71,220.00                               | Nil                  | 16,71,220.00                                 | Nil   | 16,71,220.00      | Nil                | -  |  |
| 2     | CONSUMABLES                   | 1,25,000.00     | 43,750.00 + 81,250.00* = 1,25,000.00       | Nil                  | -  | 89,422.00                                   | 89,422.00         | 35,578.00          | Nil  |  |
| 3     | CONTINGENCIES                 | 60,000.00       | 21,000.00 + 39,000.00* = 60,000.00         | Nil                  | 20,138.00                                    | 42,078.00                                   | 62,216.00**       | Nil                | Nil  |  |
| 4     | TRAVEL                        | 60,000.00       | 21,000.00                                  | Nil                  | -  | -   | -                 | 21,000.00          | 39,000.00                                    |  |
| 5     | OVERHEAD                      | 3,00,000.00     | 1,04,250.00 + 93,530.00* = 1,97,780.00     | 62,500.00            | -  | 1,23,713.00                                 | 1,86,213.00       | 9,351.00           | 1,02,220.00                                  |  |
|       | Recurring Items (GENERAL)     | 5,45,000.00     | 1,90,000.00 + 2,13,780.00* = 4,03,780.00   | 62,500.00            | 20,138.00                                    | 2,55,213.00                                 | 3,37,851.00       | 65,929.00          | 1,41,220.00                                  |  |
|       | TOTAL                         | 24,30,000.00    | 20,75,000.00                               | 62,500.00            | 16,91,358.00                                 | 2,55,213.00                                 | 20,09,071.00      | 65,929.00          | 1,41,220.00                                  |  |

NOTE: \* transferred from the Capital head (unspent amount).

\*\* An extra amount Rs. 2,216.00 spent from "Contingency" is adjusted from the "Overhead" section.

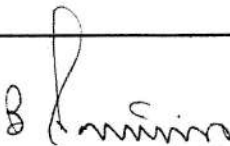
  
Signature of PI

  
Signature of Accounts Officer  
Finance Officer  
Tezpur University

  
Signature of the Head of the Institute  
Registrar  
Tezpur University

**UTILISATION CERTIFICATE**  
**(TO BE SENT IN DUPLICATE TO DEPARTMENT OF SCIENCE AND TECHNOLOGY)**  
**For all financial years 2016-17 (up to September 2016)**

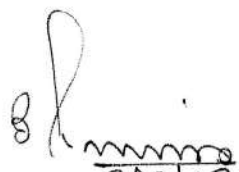
|     |  |  |
|-----|--|--|
| 1.  | Title of the Project/Scheme  | Utilization of the Calcium Carbonate obtained from Lime Sludge waste of Indian Paper Industry in the fabrication and study of mechanical properties of calcium carbonate based 'Lime Sludge filled Polyethylene' composites and Optimization of their manufacturing parameters using multi-objective Evolutionary Algorithms |
| 2.  | Name of the institution  | TEZPUR UNIVERSITY  |
| 3.  | Principal Investigator   | Satadru Kashyap  |
| 4.  | Dept. of Science and Technology sanction order no. and date of sanctioning of the project  | SB/FTP/ETA-88/2013 dated 03.09.2013  |
| 5.  | Head of the account as given in the original sanction order  | Capital (Equipment): 18,85,000.00<br>Consumables: 1,25,000.00<br>Contingencies: 60,000.00<br>Travel: 60,000.00<br>Overhead: 3,00,000.00  |
|     |  | Total : Rs 24,30,000.00  |
| 6.  | Amount brought forward from the previous financial year quoting DST letter No. and date in which the authority to carry forward the said amount was given. | Rs. 65,929.00  |
| 7.  | Amount received during the financial year (Please give DST letter/order no. and date)  | Nil  |
| 8.  | Total amount that was available for expenditure (excluding commitments) during the financial year (Sr. no. 6+7)  | Rs. 65,929.00  |
| 9.  | Actual expenditure (excluding commitments) incurred during the financial year  | Rs. 54,677.00  |
| 10. | Balance amount available at the end of the financial year  | Rs. 11,251.00  |
| 11. | Unspent balance refunded, if any (please give the details of Cheque No. etc)   | Rs. 11,252.00  |
| 12. | Amount to be carried forward to the next financial year (if applicable)  | Not applicable   |

  
**Finance Officer**  
**Tezpur University**


**UTILISATION CERTIFICATE**

Certified that the amount of Rs. 20,75,000.00 of grant sanctioned during the year 2013 -14 in favor of Registrar, Tezpur University vide sanction order no SB/FTP/ETA-88/2013 on 03.09.2013, a sum of Rupees Rs. 62,500 for year 2013-14, Rs. 16,91,358.00 for year 2014-15, Rs. 2,55,213.00 for year 2015-16 and 54,677.00 for year 2016-2017 (up to 2<sup>nd</sup> September 2016 – Project Completion date) has been utilized for the purpose of Research project for which it was sanctioned and that the balance of Rs. 11,252.00 is refunded herewith vide DD no. .... dated .....

  
(SATADRU KASHYAP)  
Name & Signature  
of PI

  
Name & Signature  
of Accounts Officer with seal

*Finance Officer  
Tezpur University*

  
Name & Signature of  
Head of the Institution  
With date and seal  
*Registrar  
Tezpur University*

**(TO BE FILLED BY DST)**

Certified that I have satisfied myself that the conditions on which the grant was sanctioned have been fulfilled/ are being fulfilled and that I have exercised the following checks to see that the money was actually utilized for which it was sanctioned.

Kinds of checks exercised

- i)
- ii)

Signature  
Designation  
Date:

**STATEMENT OF EXPENDITURE**  
2013 -14, 2014-15, 2015-16 & 2016-17 (up to Sept. 2016)

| S. No | Sanctioned Heads       | Funds Allocated | Funds Received                                   | Expenditure Incurred          |   |   |   | Total Expenditure | Amount to be returned to SERB   | Remark (if any)  |
|-------|------------------------|-----------------|--|-------------------------------|---|---|---|-------------------|---------------------------------|--|
|       |                        |                 |  | 2013-2014                     | 2014 -15<br>(till 31 <sup>st</sup> March, 2015) | 2015-16 (till 31 <sup>st</sup> March, 2016) | 2016-17 (till 2 <sup>nd</sup> Sept, 2016) |                   |                                 |  |
| 1     | CAPITAL<br>(Equipment) | 18,85,000.00    | 16,71,220.00 +<br>2,13,780.00* =<br>18,85,000.00 | Nil                           | 16,71,220.00                                    | Nil   | Nil                                       | 16,71,220.00      | Nil                             | Balanced of Rs. 2,13,780.00 transferred to Recurring heads (with permission from SERB) |
|       |                        |                 |  | Non-recurring Items (CAPITAL) | 18,85,000.00                                    | 16,71,220.00                                | Nil                                       | Nil               | 16,71,220.00                    |  |
| 2     | CONSUMABLES            | 1,25,000.00     | 43,750.00 +<br>81,250.00* =<br>1,25,000.00       | Nil                           | -   | 89,422.00                                   | 35,574.00                                 | 1,24,996.00       | 4.00                            |  |
| 3     | CONTINGENCIES          | 60,000.00       | 21,000.00 +<br>39,000.00* =<br>60,000.00         | Nil                           | 20,138.00                                       | 42,078.00                                   | Nil                                       | 62,216.00**       | - 2216.00                       |  |
| 4     | TRAVEL                 | 60,000.00       | 21,000.00  | Nil                           | -   | -   | 9,788.00                                  | 9,788.00          | 11,212.00                       |  |
| 5     | OVERHEAD               | 3,00,000.00     | 1,04,250.00 +<br>93,530.00* =<br>1,97,780.00     | 62,500.00                     | -   | 1,23,713.00                                 | 9315.00                                   | 1,95,528.00       | (1,97,780 - 1,95,528 = 2252.00) |  |
|       |                        |                 | Recurring Items (GENERAL)                        | 5,45,000.00                   | 2,13,780.00* +<br>4,03,780.00                   | 62,500.00                                   | 20,138.00                                 | 2,55,213.00       | 54,677.00                       | 3,92,528.00  |
| TOTAL |                        | 24,30,000.00    | 20,75,000.00                                     | 62,500.00                     | 16,91,358.00                                    | 2,55,213.00                                 | 54,677.00                                 | 20,63,748.00      | 11,252.00                       |  |

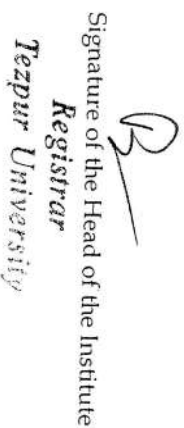
NOTE: \* transferred from the Equipment head (unspent amount of Rs. 2,13,780.00) to Consumables (Rs. 81,250.00), Contingency (Rs. 39,000.00) and Overhead expenses (Rs. 93,530.00) with permission from SERB.

\*\* An extra amount Rs. 2,216.00 spent from "Contingency" is adjusted from the "Overhead" section.

  
Signature of PI

  
Signature of Accounts Officer

  
Finance Officer  
Tezpur University

  
Signature of the Head of the Institute  
Registrar  
Tezpur University