Thermo-Mechanical Characterization of residue powder extracted from Rohu Fish Scale in Composite Material Development

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Abstract
Livestock waste is been used currently in various applications like textiles, crafting, decorations and even in biocomposite manufacturing. The reason for it is the abundance availability and compatible characteristics of the Livestock waste. Here the reviews by various literature sited has been piled up to enhance the awareness of the researchers in the area of using Livestock waste as a reinforcing material in developing various forms of composites. Properties like physical, chemical, mechanical, thermal, acoustical, morphological etc. that are been characterized by different researchers have been put together for the advancement of material development technology. In the past Importance is being given by most of the researchers to the various natural plant fibers. So for the change and the sustainable development the reviews are been formatted. The efforts are made to focus on advanced technology and uplift the use of residue powder extracted from Rohu Fish Scale as a Natural Biomaterial which is at present serve as a waste of Fish industry and to enhance the use of livestock waste in a sustainable growth of the earth and healthy environment.

Keywords — *Composites, residue powder extracted from Rohu Fish Scale, environment friendly*

I. INTRODUCTION

Composite material can be well-defined as a material system composed of two or more unlike components, insoluble in each other, differing in forms, physically distinct and chemically inhomogeneous [1]. The resulting output component possesses properties which are different as compared to individual constituent materials. Composite basically consists of a major constituent matrix and a reinforcing agent i.e. a fiber or any particulate. Now days the combination of particulate, nano-nano, nano-micro, sizing variations, orientation manipulations and thousands of other combinations are made along with the matrix in order to enrich the properties of the resultant composite material and to get the desired output based on the applications of the material.

In the current research work, the hybrid bio composite is fabricated using epoxy resin (CY-230) discovered in 1909 by Prileschajew [2] and Ciba-Geigy Ltd. in 1946 first introduced epoxy resins [3] as matrix with hardener (HY-951) as curing agent, reinforced with 5 wt% chicken feather (already optimized) and varying weight percentages (wt%) of extracted residue powder (ERP) from Rohu fish scals. Nano sized $CaCO_3$ when added to trifunctional epoxy resin shows good thermal stability and improved cross linking properties [4]. The white colored ash extracted from residue of Rohu fish is used as particulate in order to improve the mechanical properties of developed composite.

II. MATERIALS

Specifically in the present research work, the development of bio-composite requires the matrix (as the major constituent) which is CY-230 Epoxy resin, Binding agent (Hardener) which is HY-951 of 10 wt% [5-6], Reinforcing agent which is 5 wt% Chicken Feathers [7-8] and the various wt% Particulate which is extracted residue powder from Rohu fish.

The different materials used for the casting of composite are described below as shown in fig. 1.

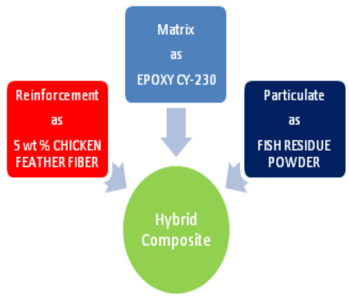

Fig. 1: Constituents in hybrid composite

A. *Extraction of Particulate Element from Rohu fish Scale*

Particulates are the fine micro or nano sized particles used in the composite development to uplift the properties of the homogeneous material. Here, the white semi crystalline micro sized powder prepared by treating the residue of Rohu fish scale under various laboratory methods is used as a particulate in the development of biocomposite. The prepared powder contains various elements as seen through Field Emission Scanning Electron Micrography (FE-SEM) and XRD analysis has been done.

ERP is the white colored micro sized particle which was extracted from the Rohu fish scale. The various laboratory reactions were performed in the development of ERP. The developed ERP contains varying percentages of elements like calcium, sodium, and oxygen etc. in compound form which shows better compatibility with Epoxy.

The white powdered particulate was extracted from Rohu fish scale using systematic step by step processing as the silica is extracted from rice husk ash[9]. The sequential process is described below and the pectoral view of the complete process is shown in fig. 2.

- Skin of rohu (Labeo rohita) with the size of 1.0-2.0 kg was obtained from a college of fisheries, G B Pant University of Agriculture & Technology Pantnagar, India. The residual meat on the skin was removed by knife and washed with distilled water in the lab.
- Take the residue in a metallic beaker and burn it in open atmosphere at around 2000C till it completely burns (black colored and oily appearance). Open atmosphere is recommended as burning of fish deposite makes pungent and foul smell.
- Try to remove maximum amount of black oil from the burnt fish residue by absorbing sheet and filter paper to get dry black substantial (as much as possible) and keep it to dry for 48 hours approximately. (Black semi solid paste type appearance can be seen).
- Treat the oily ash mixture with thrice the weight of NaOH and heat it at 1800C for 1 hour in closed oven. Use magnetic stirrer and stir the mixture at 1800C at approximately 450 rpm for 2 hours.
- With the help of glass dropper add H2SO4 (sulphuric acid) drop wise and be alert as it makes accidental chances while hydrogen liberation takes place. Keep the sample for 5 hours at rest in atmospheric condition.
- Now heat the substance for 1 hour each in a high temperature furnace with the hit and trial method at various temperatures starting from 3000C than 4000C than 5000C- 6000C etc. till the desired appearance is visible and keep observing the color change.
- At around 7200C you will observe the white crystalline solid similar to silica. Check at validate result via TGA/DTA and other analysis.
- By the composition detection (using FE-SEM) the clear view regarding the composition of extracted powder was recovered and it was found that apart from silica the extracted residue powder contains calcium, sodium, phosphorous, etc. in elemental and CaCO3, SiO2 etc. in compound form.
- The variation that occurred during the entire process was captured and is shown in figure 2.
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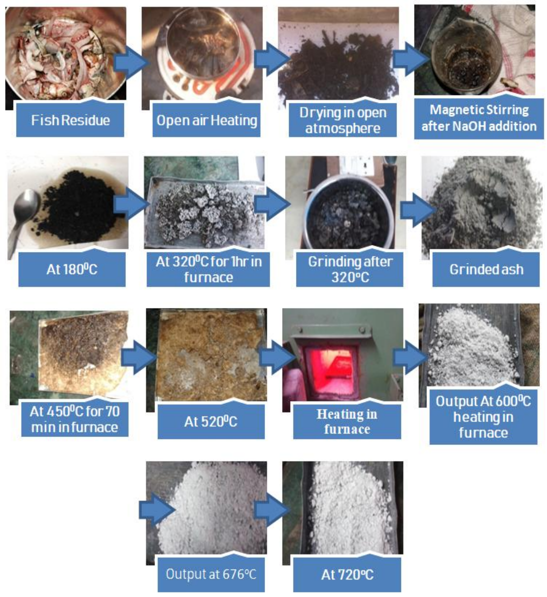

Fig. 2: Step by step processing performed during extraction of fish residue powder

III. RESULTS AND DISCUSSIONS

The systematic fabrication and processing of various composite's samples with varying weight percentages residue powder extracted from Rohu Fish Scale in epoxy resin had led us to are based on the results of mechanical tests performed at the Dynamics Lab, Collage of Technology, G. B. Pant University of Agriculture and Technology, Pantnagar.

The variation in density and volume void fraction in hybrid composite with increasing wt% of extracted residue powder as shown in fig. 3.

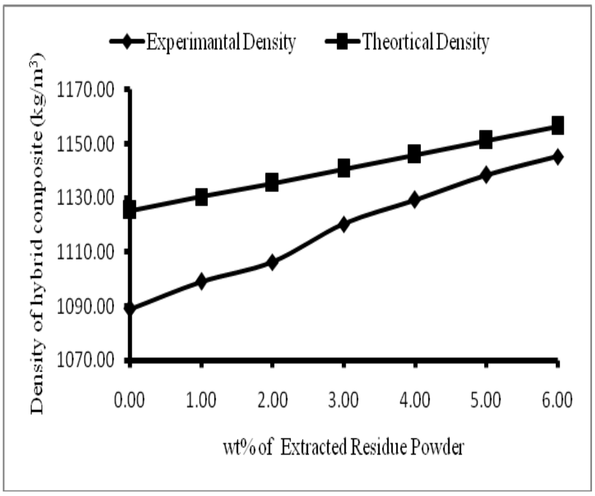

Fig. 3: Effect of wt% of extracted residue powder (in 5 wt% CFF) on density of composite

Varying wt% of extracted residue powder was added in the resin matrix and it was interesting to note that the weight density of bio composite (figure 3) was continuously rising as the wt% of ERP was increased. It was 1089.12 kg/m3 at 0 wt% of ERP and raises to 1145.45 kg/m3 at 6 wt% of ERP in composite. The rise of 5.17 % is monitored during the experimentation from 0 to 6 wt%.

TABLE I

Density and volume void fraction percentage for various compositions of ERP and 5 wt% CFF filled epoxy resin hybrid composite

Compos -ition	Experimental Density (kg/m3)	Theoreti -cal Density (kg/m3)	Volume Void Fraction (%)
ERP0 = 0 wt% ERP	1089.12	1125.38	3.22
ERP1= 1 wt% ERP	1099.10	1130.47	2.78
ERP2= 2 wt% ERP	1106.36	1135.61	2.58
ERP3= 3 wt% ERP	1120.46	1140.79	1.78
ERP4= 4 wt% ERP	1129.44	1146.02	1.45
ERP5= 5 wt% ERP	1138.53	1151.30	1.11
ERP6= 6 wt% ERP	1145.45	1156.63	0.97

The rise in density may be due to the upright attraction bonding between the matrix and the particles at 3 wt% of ERP in 5 wt% CFF filled composite which can be clearly seen in SEM image shown in fig. 4. The decrease in volume void fraction with inclusion of ERP as given in table 1 depicts the improved strength and adhesion bonding in the hybrid composite.

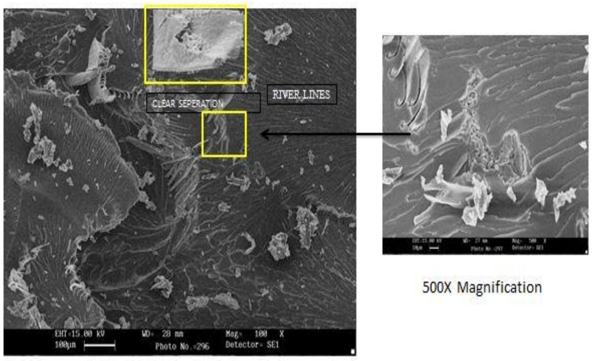

Fig. 4: SEM image of hybrid composite with 1 wt% extracted residue powder in epoxy resin at 100X magnification

Fig. 5-6 shows the results obtained using 25 kN servo controlled Universal Testing Machine (UTM) for tensile testing. The fixed cross head speed of 1mm/min was selected for the generation of load v/s displacement curve.

Here different wt% of ERP in 5 wt% CFF filled hybrid bio-composite samples were tested and the obtained stress/strain relation is shown in figure 5.

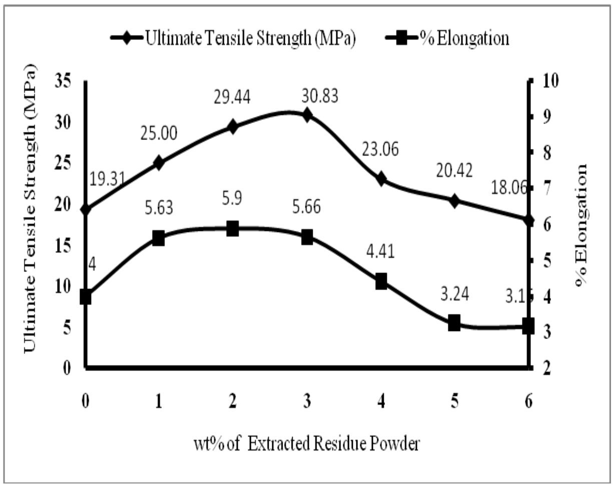

Fig. 5: Effect of ERP wt% (with 5 wt% CFF) on ultimate tensile strength and percentage elongation on hybrid composite

The maximum tensile strength (i.e. 30.83MPa) was observed at 3 wt% ERP which is 70.71% greater than 0 % ERP sample. Also the percentage elongation was rising with increasing wt% of ERP till 3% (% elongation at 3% ERP was 5.78). Later with increasing wt% of ERP, percentage elongation decreases down to 3.5% which shows its brittle characteristic as shown in 6.

TABLE II

Young modulus and reliability factor (R2 value) of various hybrid composites with extracted residue powder as particulate in 5 wt% CFF filled epoxy resin

wt% of ERP	Young Modulus (MPa)	R2 Value
0	800.85	0.9994
1	667.88	0.9997
2	666.08	0.9997
3	1115.8	0.9999
4	804.86	0.9998
5	855.33	0.9996
6	775.63	0.9993

The increase in the young modulus 6 and ultimate tensile strength 5 might be due to the better interfacial bonding and matrix- reinforcement cross linking as shown in SEM micrograph. This also enhanced the toughness characteristics of the hybrid composites.

The thermal analyses of the prepared samples were diagnosed using TGA/DTA analysis at the IIC

Laboratory, IITR. Figure 7-12 shows the rate of decomposition and variation in the specimen with temperature and time through DTA, DTG and TG curve of the hybrid composite samples performed in Air environment.

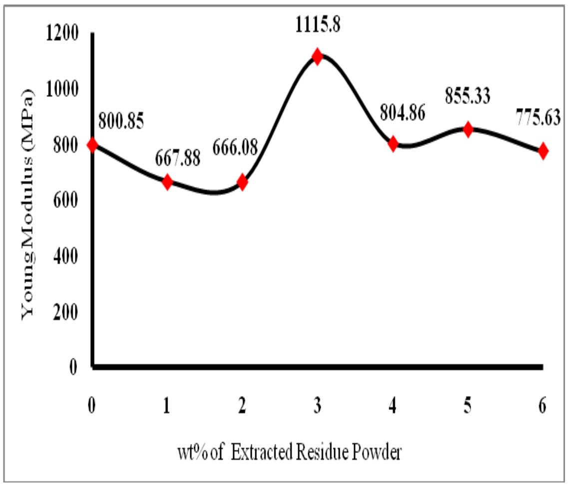

Figure 6: Effect of ERP wt % on young modulus of hybrid composite

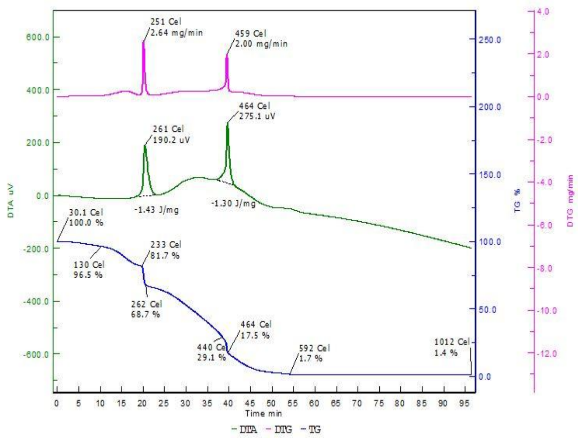

Fig. 7: Thermal analysis of 1% extracted residue powder + 5% CFF in epoxy resin hybrid composite

Fig. 7 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 1 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.210 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 251°C to 459°C with corresponding rate of decomposition ranging 2.64 mg/min to 2.0 mg/min. Prior to 233°C, the weight loss of 18.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 2.64 mg/min was observed at 251°C. Such decomposition has been supported with the heat of fusion of -1.43 J/mg at 261°C and -1.30 J/mg at 464°C with DTA signal of 190.2 µV (0.1902mV) to 275.1 µV (0.2751mV) between temperature range of 261°C and 464°C respectively. The decomposition of the material had been concluded at about 592°C leaving carbonize residue 1.7% of initial weight.

Fig. 8 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 2 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.18 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 262°C to 426°C with corresponding rate of decomposition ranging 4.3 mg/min to 3.10 mg/min. Prior to 239°C, the weight loss of 15.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.3 mg/min was observed at 262°C. Such decomposition has been supported with the heat of fusion of -2.06 J/mg at 277°C and -3.85 J/mg at 452°C with DTA signal of 265 µV (0.265 mV) to 648 µV (0.648 mV) between temperature range of 277°C and 452°C respectively. The decomposition of the material had been concluded at about 518°C leaving carbonize residue 2.2% of initial weight.

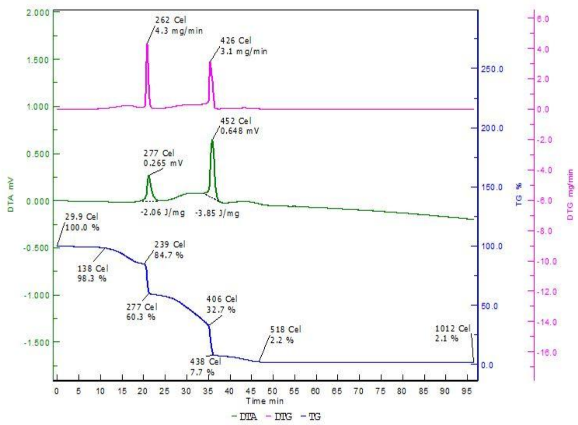

Fig. 8: Thermal analysis of 2% extracted residue powder + 5% CFF in epoxy resin hybrid composite

Fig. 9 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 3 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.32 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 262°C to 425°C with corresponding rate of decomposition ranging 4.4 mg/min to 2.90 mg/min. Prior to 239°C, the weight loss of 15.60% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.4

mg/min was observed at 262°C. Such decomposition has been supported with the heat of fusion of -1.91 J/mg at 278°C and -3.65 J/mg at 450°C with DTA signal of 272 µV (0.272 mV) to 633 µV (0.633 mV) between temperature range of 278°C and 450°C respectively. The decomposition of the material had been concluded at about 522°C leaving carbonize residue 2.9% of initial weight.

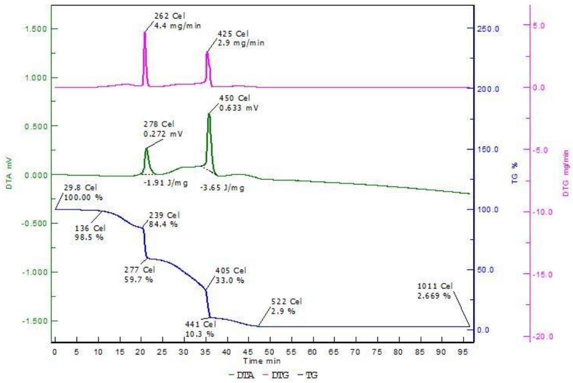

Fig. 9: Thermal analysis of 3% extracted residue powder + 5% CFF in epoxy resin hybrid composite

Fig. 10 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 4 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.27 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 258°C to 422°C with corresponding rate of decomposition ranging 5.0 mg/min to 3.0 mg/min. Prior to 245°C, the weight loss of 16.20% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 5 mg/min was observed at 258°C. Such decomposition has been supported with the heat of fusion of -2.33 J/mg at 283°C and -4.89 J/mg at 446°C with DTA signal of 306 µV (0.306 mV) to 724 µV (0.724 mV) between temperature range of 283°C and 446°C respectively. The decomposition of the material had been concluded at about 505°C leaving carbonize residue 0.9% of initial weight.

Fig. 11 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 5 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.20 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 257°C to 409°C with corresponding rate of decomposition ranging 4.3 mg/min to 3.2 mg/min. Prior to 240°C, the weight loss of 15.30% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve.

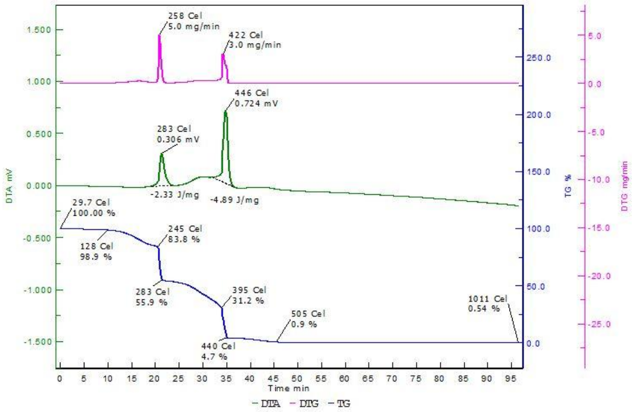

Fig.10: Thermal analysis of 4% extracted residue powder + 5% CFF in epoxy resin hybrid composite

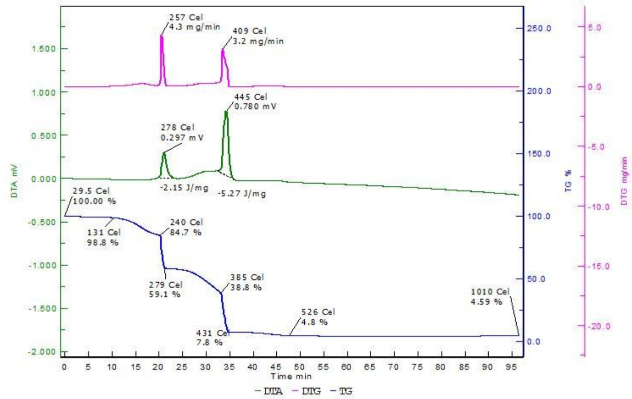

Fig.11: Thermal analysis of 5% extracted residue powder + 5% CFF in epoxy resin hybrid composite

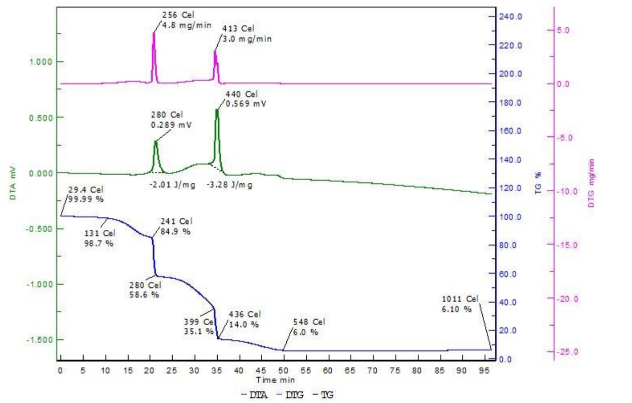

Fig.12: Thermal analysis of 6% extracted residue powder + 5% CFF in epoxy resin hybrid composite

The maximum rate of decomposition of 4.3 mg/min was observed at 257°C. Such decomposition has been supported with the heat of fusion of -2.15 J/mg at 278°C and -5.27 J/mg at 445°C with DTA signal of 297 µV (0.297 mV) to 780 µV (0.780 mV) between temperature range of 278°C and 445°C respectively. The decomposition of the material had been concluded at about 526°C leaving carbonize residue 4.8% of initial weight.

Fig. 12 shows Thermo Gravimetric Graph of Epoxy resin (CY-230) based hybrid composite with 10 wt% Hardener (HY-951), 5 wt% CFF and 6 wt% Extracted Residue Powder from fish residue. The initial sample weight was 10.18 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 256°C to 413°C with corresponding rate of decomposition ranging 4.8 mg/min to 3.0 mg/min. Prior to 241°C, the weight loss of 15.10% may be attributed to the expulsion of the moisture, low molecular mass molecules and volatile matter associated with the material which can be observed in TG curve. The maximum rate of decomposition of 4.8 mg/min was observed at 256°C. Such decomposition has been supported with the heat of fusion of -2.01 J/mg at 280°C and -3.28 J/mg at 440°C with DTA signal of 289 µV (0.289 mV) to 569 µV (0.569 mV) between temperature range of 280°C and 440°C respectively. The decomposition of the material had been concluded at about 548°C leaving carbonize residue 6% of initial weight.

IV. CONCLUSIONS

Fabrication and thermo-mechanical characterization of epoxy based composites was performed in the present research work. Among all the Epoxy Resin based composites developed with varying weight percentage of chicken feather fiber reinforcement i.e. 0 wt% (control sample) to 7 wt% of CFF, the 5 wt% of CFF in cured epoxy resin was the most appropriate composition based on the output results of strength and for hybrid biocomposite development with extracted powder from Rohu fish by varying wt% of particulate from 0 wt% (i.e. 5 wt% CFF) to 6 wt% of reinforcing particulate the 3 wt% of particulate gave the most optimized results during the entire investigation of hybrid composite characterization. The depiction of mechanical properties is done through tensile test. The thermal analysis is done using TGA/DTA. The entire paper helps to get the deep knowledge about the material used, methods adopted, process required for complete development of material, its testing procedure and the systematic standardization.

With the rise in wt% of particulate (extracted residue powder from fish) the density of the hybrid composite rises and it reaches close to density of cured epoxy resin at 3 wt% of ESP in 5 wt% CFF filled composite. The enhanced mechanical properties were detected with improved tensile 3 wt% of Extracted Powder as particulate reinforcement. The thermal analysis of hybrid composites (1 to 6 wt% ESP) was performed using output graphical representation of TGA/DTA Curve. The separate DTA, DTG and TG curve for every sample gave the complete information about the decomposition rate, thermal characteristics and thermal stability. The 1 wt% ESP reinforced hybrid composite showed maximum burying temperature of 5920C for complete combustion. The initial decomposition temperature and time required for complete concealing of the composite was spotted through the exploration.

REFERENCES

[1] Gupta, K.M. 2006. Material Science, Second Edition, Umesh Publications, Ch-17, Other Material (wood, concrete, glass elastomer, composites etc.). 1: 428-431.

[2] May, C.A. 1988. Epoxy Resins, Chemistry and Technology, Second Ed., Marcel Dekker, New York.

[3] Nagarjuna, R.P., Smita, M. and Nayak, S.K. 2014. Synthesis and Modifications of epoxy Resins and their Composites: A Review. In Taylor and Francis, Polymer-Plastics Technology and Engineering, 53:16, 1723-1758.

[4] Fan-Long, J. and Soo-Jin, P. 2009. Thermal Stability of Tri functional Epoxy Resins Modified with Nanosized Calcium Carbonate. Bull. Korean Chem. Soc. 30: 334-338.

[5] Singh, V.K. and Gope, P.C. 2010. Silica-Styrene-Butadiene Rubber Filled Hybrid Composites. Experimental Characterization and Modeling. Journal of Reinforced Plastics and Composites. 29: 2450-2468

[6] Singh, V.K., Bansal, G., Agarwal, M. and Negi, P. 2016. Experimental Determination of Mechanical and Physical Properties of Almond Shell Particles Filled Biocomposite in Modified Epoxy Resin. Journal of Material Science & Engineering ISSN: 2169-0022 Issue 3. Volume 5.

[7] Kumar A, Bansal G, Singh V. K. “Characterization of Mechanical Strength of Epoxy Hybrid Composite Reinforced with Chicken Feather Fiber and Residue Powder Extracted from Rohu Fish Scale.” International Journal of Engineering Research & Technology (IJERT) ISSN: 2278-0181, Vol. 8 Issue 04, pp. 498-504.

[8] Narendra, R., Jiasong, J. and Yiqi, Y. 2014. Biodegradable Composites Containing Chicken Feathers as Matrix and Jute Fibers as Reinforcement. Springer Journal, DOI 10.10 07/s 10924-014-0648-9. 310-318.

[9] Mittal, D. 1997. Silica from Ash-A valuable product from waste material. General Article, Sant Longowal Institute of engineering and Technology. pp. 64-67.