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Thermal Characterization, Compositional Analysis and Extraction of Elemental Powder from Rohu Fish Residue used as Composite Particulate

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Abstract
The efforts were made for the laboratory preparation of useful elemental based powder from the waste/residue of Rohu fish. The concept was initially applied similar to the process of extracting silica from ash. Extracted Residue Powder (ERP) thus obtained by systematic processing of fish residue contained many noble elements like Silica, Calcium, Carbon, Magnesium, Sodium etc. which were scrutinized later by FESEM compositional analysis. The ERP was used as a particulate in the epoxy based composite where it shows appreciable compatibility, bonding characteristics and enhancement in mechanical properties. The extracted residue powder resulted in good thermal and decomposition characteristics as seen in TGA analysis at 320 and 720 degree centigrade.

Keywords- Rohu Fish, Epoxy Resin, Particulate, Composite, Chicken Feather Fiber.

1. Introduction

Various elements and compounds like Silica, Calcium Carbonate and Magnesium etc. were studied as a particulate having good compatibility with epoxy resin matrix (Paluvai et al., 2014). Efforts were made to extract the elements from livestock waste for the cost effective development of composite with high strength to weight ratio. With the time it was diagnosed using literature survey that the fish residue may contains many useful elements if chemically treated and processed. Jin and Park (2009) described the thermal stability of nano sized calcium carbonate ($CaCO_3$) in modified tri functional epoxy resin. The results showed high cross linking density and decreased coefficient of thermal expansion at the region of high contamination of particles. Mittal (1997) performed the laboratory experiment on rice husk ash to get silica from ash. Rice husk ash contains 90-98% silica. He then performed the following two reactions under controlled condition to get silica from ash.

$$\underset{\text{(ash)}}{SiO_2} + \underset{\text{(caustic soda)}}{2NaOH} \xrightarrow{180-200^{\circ}C} \underset{\text{(sodium silicate)}}{Na_2SiO_3} + \underset{\text{(water)}}{H_2O}$$

6-8 atm

$$\underset{\text{(sodium Silicate)}}{Na_2SiO_3} + \underset{\text{(sulphuric acid)}}{H_2SO_4} \xrightarrow{90-100^{\circ}C,1\ atm} \underset{\text{(silica)}}{SiO_2} + \underset{\text{(sodium sulphate)}}{Na_2SO_4} + \underset{\text{(water)}}{H_2O}$$

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Kalapathy et al. (2000) gave the detailed information about the process of preparation silica powder from rice husk. The results obtained were verified and justified using FTIR and XRD analysis. Zheng et al. (2009) studied thermo-mechanical properties such as glass transition temperature (Tg), dynamic mechanical analysis, tensile and flexural strength. The properties of conventional epoxy/micro-silica composite were improved by the addition of nano silica and it was due to the increase of compaction via even dispersion of the nano-silica among the micro-silica particles. The storage modulus, flexural strength etc. were determined.

So, the fish residue like bones, shells and fins were chemically treated and the powder thus obtained was checked using Field Emission Scanning Electron Microscopy compositional analysis and through Thermo Gravimetry Analysis (TGA) for the morphological and thermal analysis (Singh et al., 2016). Results thus obtained were then interpreted.

2. Procedure for Extracting Valuable ELEMENTS form Fish Residue

The white powdered particulate was extracted from Rohu fish using systematic step by step processing as the silica is extracted from rice husk ash (Mittal, 1997). The sequential process is described below and the pectoral view of the complete process is shown in Figure 1.

- Firstly, collect the waste Rohu fish fins, skin and shells in a beaker and wash it properly with the running water, then dry the deposit in sun light for 30 min (optional).
- Take the residue in a metallic beaker and burn it in open atmosphere at around 200^{o}C till it completely burns (black colored and oily appearance). Open atmosphere is recommended as burning of fish deposit 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 180^{0}C for 1 hour in closed oven. Use magnetic stirrer and stir the mixture at 180^{0}C at approximately 450 rpm for 2 hours.
- With the help of glass dropper add H_2SO_4 (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 methods at various temperatures starting from 3000C then 4000C then 5000C - 6000C etc. till the desired appearance is visible and keep observing the color change.
- At around 720^{0}C you will observe the white crystalline solid similar to silica. Check at validate result via TGA/DTA, XRD 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 $CaCO_3$, SiO_2 etc. in compound form.

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The variation that occurred during the entire process was captured and is shown in Figure 1.

3. Field Emission Scanning Electron Microscopy (FE-SEM)

Field Emission Scanning Electron Microscope (FESEM) is a microscope that works with electron (i.e. negatively charged ions). It provides topographical as well as elemental information about the entire or fractioned object under investigation in various magnifications ranging from 2X to 3,00,000X and with unlimited virtual depth of cut (Senoz et al., 2012). As compared to SEM it is much more superior and provides clearer, least distorted images with spatial resolution down to 1 ½ nanometers. FESEM can also be used as a tool that can provide information about nano size particle. Therefore, through FESEM we can get the elaborative information about the structure, composition, morphology, uniformity determination and small contamination in the material.

From Figure 2 (a-d) variations in the composition and the elements in the extracted residue powder from fish can be observed. These elements are in different weight fractions as listed in Table 1-4 which are taken at different positions of the powdered sample. Here the percentage of oxygen was found to be maximum which was later reduced using reduction techniques. Also the appreciable percentages of Calcium, Silica, Sodium, Phosphorous etc. proved the usability of ERP as a particulate material for hybrid composite manufacturing.

4. Thermal Analysis

For the current research thermal analysis TG analyzer EXSTAR TG/DTA 6300 is used at IIT Roorkee. The DTA, DTG and TG curve corresponding to change in time for ERP is graphically represented in Figures 3 (a-b). The analysis is performed in airy atmosphere with flow rate of 200mL/min and temperature rate of 10^0C/ min from 0^0C to 1000^0C.

Thermo Gravimetric Analysis is a thermal analysis method that provides information about the change in physical and chemical properties of the material as a function of temperature (with constant heat transfer), or as a function of time (with constant mass flow or temperature i.e. Isothermal mode) under control atmosphere (Singh et al., 2016). It is also represented in graph as Derivative Thermo Gravimetric curve (DTG curve).

Figure 3(a) shows Thermo Gravimetric Graph of Extracted Residue Powder from fish residue that was heat treated till 320^0C in a furnace during preparation. The initial sample weight taken for analysis was 10 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 336°C to 457°C with the corresponding rate of decomposition ranging from 0.35 mg/min to 0.92 mg/min. The maximum rate of decomposition was observed at 457^0C (Tuna et al., 2015). Also the decomposition was supported with the heat of fusion of 427 mJ/mg at 343^0C with DTA signal of 30.4 µV and 3.86J/mg at 461^0C with DTA signal of 317.3 µV. The decomposition at 320^0C was rising very fast as it was the unstable stage for the ERP. So the prepared sample

was not prominent at 320^0C preparation and needs further heating to get stable and highly acceptable powder (Bansal et al., 2016).

Figure 3(b) shows Thermo Gravimetric Graph of Extracted Residue Powder from fish residue that was heat treated till 720^0C in a furnace during preparation. The initial sample weight was 10 mg. Through DTG curve we can observe that the decomposition of this material has been accomplished under two stages ranging from 116°C to 846°C with corresponding rate of decomposition ranging 0.016 mg/min to 0.056 mg/min. It was observed that the ERP sample can sustain huge temperature that is it can be heated upto 1000^0C with only 10.08% material decomposition which may be due to volatile impurities, moisture content etc. Such decomposition has been supported with the heat of fusion of 87.2 J/mg at 848°C with DTA signal of 149.1 µV (0.1491 mV). The decomposition at 720^0C was less than 3% which shows the great thermal stability of ERP sample and its ability to withstand at higher temperature (Agarwal, 2014).

5. Conclusion

Based on the experimental results and FE-SEM analysis it was interesting to note that the extracted residue powder from fish disposable waste contains many important micro sized elements like calcium, sodium, silica, phosphorous, etc. These elements have good compatibility in hybrid composite manufacturing and can be a good reinforcing agent as particulate (Bansal and Singh, 2016).

The thermal analysis results gave the exact decomposition rate, its variation with varying temperature, rate of fusion, etc. that proved the acceptable prepared sample. The powder obtained at 720^0C was considered as the optimum micro sized particulate that is been used to hybrid various biocomposite.

The prepared ERP can be used as reinforcing agent in various composite manufacturing processes for developing cost effective and biocomposite. Its compatibility was also found with epoxy resin in the recent research works with chicken feather as fiber and ERP as particulate and the results diagnosed were quite appreciable.

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Figure 1. Step by step processing performed during extraction of fish residue powder

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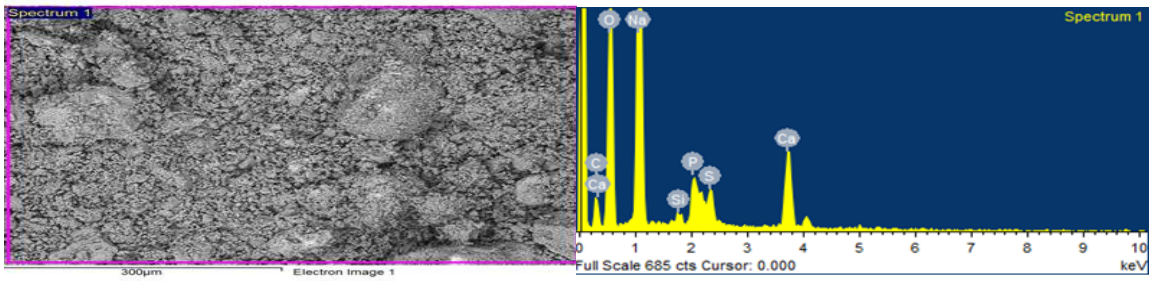

Figure 2 (a). FESEM image and composition analysis of extracted residue powder at spectrum 1 with 685 cts

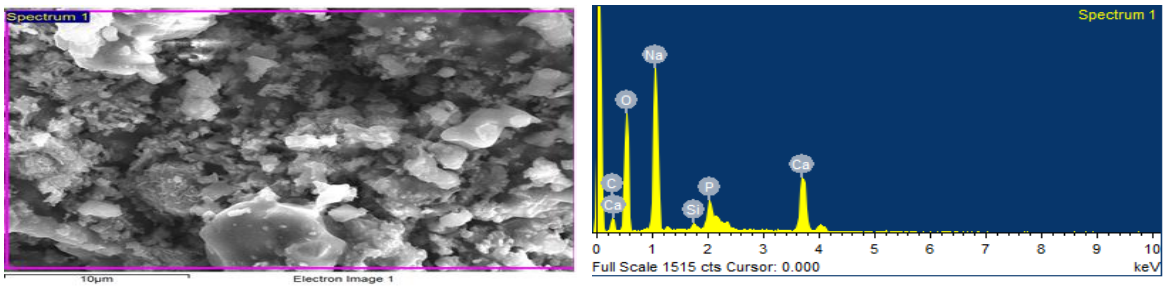

Figure 2 (b). FESEM image and Composition analysis of extracted residue powder at spectrum 1 with 1515 cts

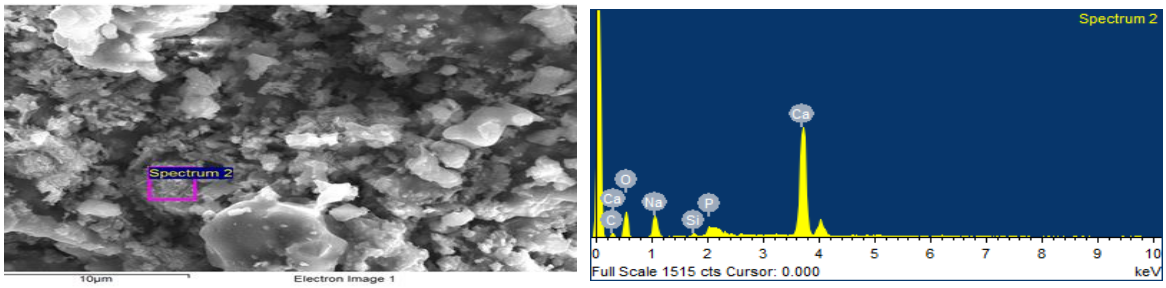

Figure 2 (c). FESEM image and composition analysis of extracted residue powder at spectrum 2 with 1515 cts

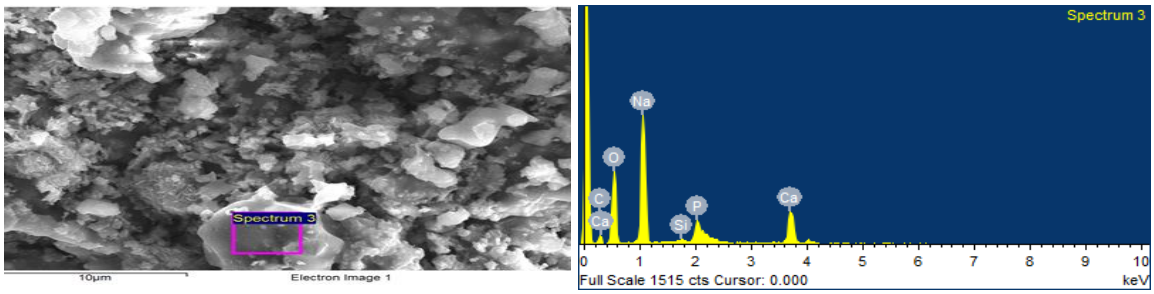

Figure 2 (d). FESEM image and composition analysis of extracted residue powder at spectrum 3 with 1515 cts

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Element	Weight%	Atomic%
C	8.86	14.05
O	44.73	53.62
Na	24.00	19.89
Si	2.78	2.53
P	4.20	2.58
Ca	15.43	7.33
Total	100.00	

Table 1. ERP composition at spectrum 1 (through FESEM at 685 cts)

Element	Weight%	Atomic%
C	4.43	14.46
O	46.40	53.72
Na	28.71	23.00
Si	7.61	2.40
P	2.22	1.32
S	1.90	1.09
Ca	8.72	4.01

Table 2. ERP composition at spectrum 2 (through FESEM at 1515 cts)

Element	Weight%	Atomic%
C	2.82	5.65
O	34.33	52.60
Na	6.36	6.65
Si	2.69	2.59
P	1.37	1.07
Ca	52.42	31.44
Total	100.00	

Table 3. ERP composition at spectrum 2 (through FESEM)

Element	Weight%	Atomic%
C	4.16	16.10
O	40.59	48.67
Na	28.45	23.55
Si	8.47	2.32
P	4.63	2.85
Ca	13.70	6.50
Total	100.00	

Table 4. ERP composition at spectrum 3 (through FESEM)

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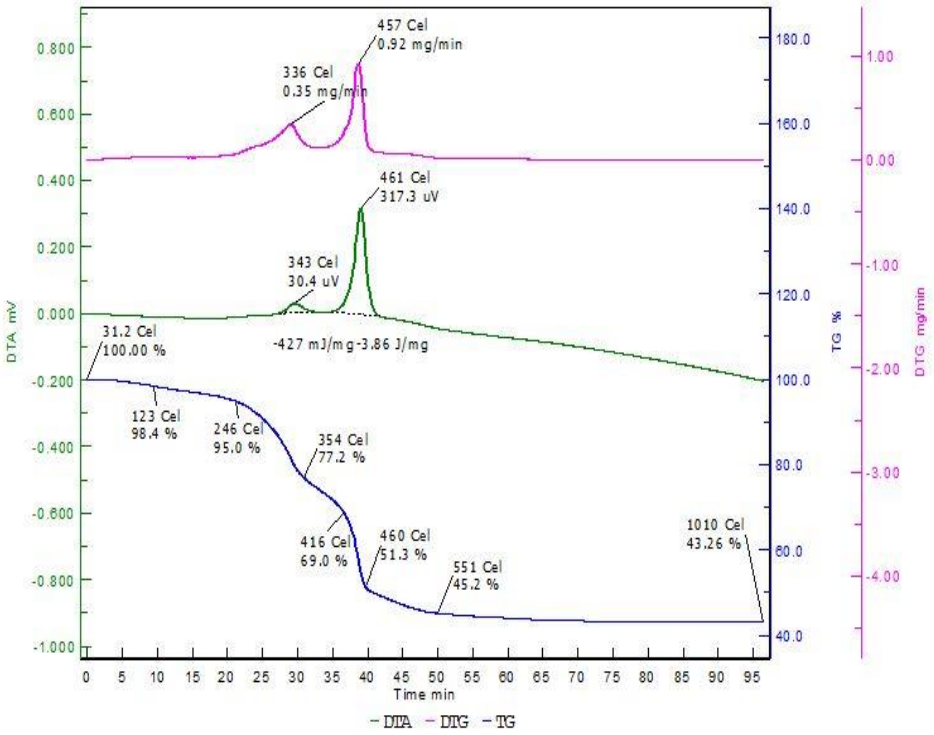

Figure 3 (a). Thermal analysis of extracted residue powder sample heated till 320^{0}C

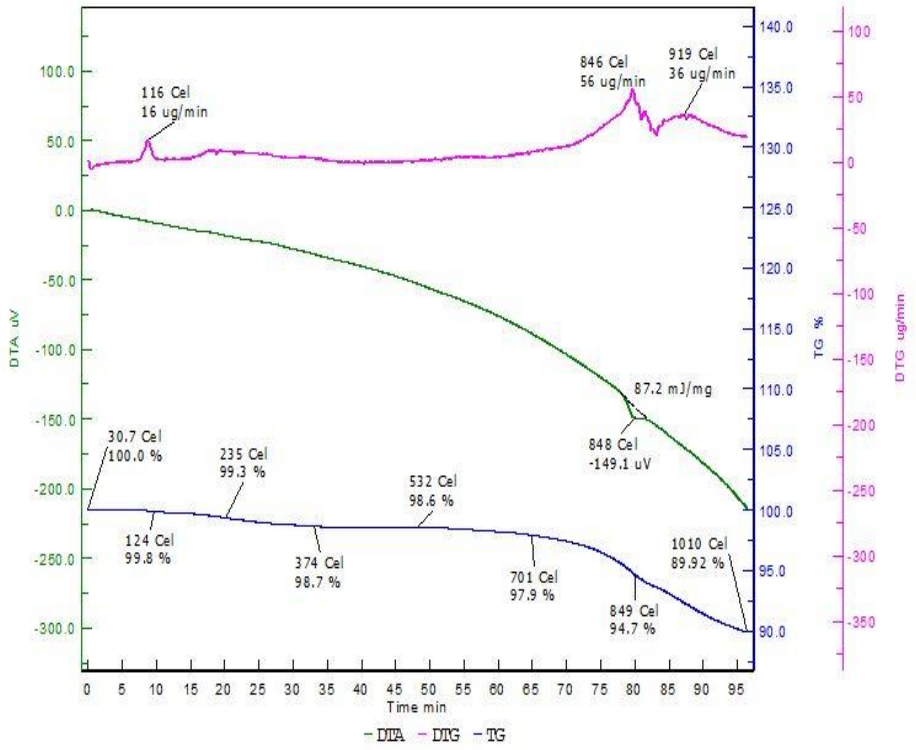

Figure 3 (b). Thermal analysis of extracted residue powder sample heated till 720^{0}C

References

Agarwal, M. (2014). Thermo-Mechanical characterization of almond shell particles filled biocomposite in modified epoxy resin. Thesis, M. Tech. G. B. Pant University of Agriculture and Technology, Pantnagar.

Bansal, G., & Singh, V. K. (2016). Flame retardation characterization of chicken feather fiber and extracted residue powder from fish. International Journal of Engineering Science, 2579-2581.

Bansal, G., Singh, V. K., Patil, P., & Rastogi, S. (2016). Water absorption and thickness swelling characterization of chicken feather fiber and extracted fish residue powder filled epoxy based hybrid biocomposite. International Journal of Waste Resources, doi:10.4172/2252-5211.1000237, 6(3), 1-6.

Jin, F. L., & Park, S. J. (2009). Thermal stability of trifunctional epoxy resins modified with nanosized calcium carbonate. Bull Korean Chemical Society, 30(2), 334-338.

Kalapathy, U., Proctor, A., & Shultz, J. (2000). A simple method for production of pure silica from rice hull ash. Bioresource Technology, 73(3), 257-262.

Mittal, D. (1997). Silica from Ash-A valuable product from waste material. General Article, Sant Longowal Institute of Engineering and Technology, 2(7), 64-66.

Paluvai, N. R., Mohanty, S., & Nayak, S. K. (2014). Synthesis and modifications of epoxy resins and their composites: A Review. Polymer-Plastics Technology and Engineering, 53(16), 1723-1758.

Senoz, E., Wool, R. P., McChalicher, C. W., & Hong, C. K. (2012). Physical and chemical changes in feather keratin during pyrolysis. Polymer Degradation and Stability, 97(3), 297-307.

Singh, V. K., Bansal, G., Agarwal, M., & Negi, P. (2016). Experimental determination of mechanical and physical properties of almond shell particles filled bio composite in modified epoxy resin. Journal of Material Science and Engineering, 5(3), 2169-0022.

Singh, V. K., Bansal, G., Negi, P., & Bisht, A. (2016). Characterization of flexural and impact strength of jute/almond hybrid bio composite. Journal of Testing and Evaluation, DOI: 10.1520/JTE20150414, 45(3), 1-11.

Tuna, A., Okumuş, Y., Çelebi, H., & Seyhan, A. T. (2015). Thermochemical conversion of poultry chicken feather fibers of different colors into microporous fibers. Journal of Analytical and Applied Pyrolysis, 115, 112-124.

Zheng, Y., Chonung, K., Wang, G., Wei, P., & Jiang, P. (2009). Epoxy/nano-silica composites: Curing kinetics, glass transition temperatures, dielectric, and thermal–mechanical performances. Journal of Applied Polymer Science, 111(2), 917-927.