# STUDIES OF MECHANICAL PROPERTIES OF BOTTOM ASH FILLED PBT COMPOSITES

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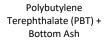
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## Article history

Received 17 June 2024 Received in revised form 15 November 2024 Accepted 31 January 2025 Published online 31 August 2025

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# **Graphical abstract**





Twin Screw Extruder



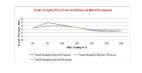


Test Specimens



Injection Molding Machine





Results

## **Abstract**

Particulate filled polymer composites have been very useful due to their low cost and wide engineering applications. Fly Ash as well as Bottom Ash emerging from thermal power plants have been an inevitable environmental hazard. Polybutylene Terephthalate (PBT) polymer has outstanding mechanical, electrical, chemical properties, better dimensional stability and low moisture absorption properties. Therefore, it has been very widely used for automobile and industrial applications. PBT composite of Bottom Ash, with and without a coupling agent has been made in this work. It is found that flexural modulus and flexural strength of this composite have increased for 90 microns Bottom Ash with the 15% (by wt%) loading and also a cost saving of 15% is reported. However tensile strength and impact strength are found to be decreasing marginally with the increase in % loading of Bottom Ash in the polymer. Scanning electron microscope (SEM) was used to characterize the samples for microstructure.

Keywords: Bottom Ash, Untreated Bottom Ash (UBT) PBT composite, Scanning electron microscope (SEM), Fly ash.

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## 1.0 INTRODUCTION

Jambhulkar et al., have enunciated that fly ash has been an inevitable environmental hazard across the globe. Lots of fly ash is generated in thermal power plants because of burning of coal and it is predicted that in coming years around 230 million tons of fly ash will be produced. Fly ash is the result of burning

of coal in boilers and is carried by the hot flue gases along with heat. In India fly ash generation is very high and therefore its management is of utmost importance. About 40% of the fly ash is unutilized [1]. Hagemeyer et al., have presented that fly ash is a part of coal ash. Coal combustion residuals (CCR), consist of small particles of radioactive materials, aluminum, arsenic, iron, lead, and mercury. Coal ash is a pollutant and it is

reported that exposure to coal ash increases chances of respiratory diseases [2]. As per Muhardi et al., coal is used as fuel in thermal power plants. Combustion of coal produces different types of coal ash namely fly ash, bottom ash, boiler slag, clinker etc. [3]. As per Bajare et al., Singh and Siddique, Menéndez et al., the coal ash which is heavy and hard cannot be carried away by flue gases, settles at the bottom of the furnace. It is called coal bottom ash [4] [5] [6]. Penaloza has enunciated that bottom ash is an environmental hazard and need to be disposed. The addition of inorganic fillers to polymers, result in a composite material which shows enhanced physical properties than that of the virgin polymers and conventional composite counterparts [7]. Bicer developed a composite of fly ash and fragmented polypropylene (PP). As the fly ash rate increases, samples produced at 225°C have exhibited better thermal properties. Mechanical properties are found to be enhanced with high PP ratio and high production temperature [8]. Lirer et al., have devised composite made of dredged sediments in a fly-ash based geo polymer. The results have shown that dredged sediments with fly ash can lead to geo polymeric matrices with better mechanical properties [9]. Zhang et al., have substituted fly ash by agricultural waste-rice husk ash (RHA) in cementitious composites (ECC). It is found that replacing fly ash by RHA, tensile properties of ECC mixtures are improved [10]. Kasar et al., have established that uniform distribution of the Fly Ash (FA) in Al matrix composite enhances its compressive strength and hardness, however it has also found that tensile strength and ductility has reduced to some extent [11]. Sim et al., have devised a fly ash/epoxy composite. It has been found that tensile strength has increased with the increase in percentage of fly ash [12]. Kastiawan et al., have researched the effect of holding time and bottom ash particle size on the properties of composites of polypropylene. The study has established that the particle size of 250-300 mesh gives the highest composite strength [13]. Shivakumar et al., have established that ash composites made from polyurethane and fly ash can decrease leaching of coal ash in-organics [14]. Dey and Pandey have found that addition of fly ash particle in to the metallic matrix enhances ultimate tensile strength hardness and wear resistance of the composite [15]. Menéndez et al., have studied ground coal bottom ash and coal fly ash cements for its pozzolanic properties to assess their pozzolanic performance [16]. Chan et al., have reviewed the properties of wollastonite-filled polymer composites [17]. Adams et al., have devised a method which is cost-effective to prepare wollastonite/PVA composites [18]. Esmailpour et al., have studied effects on fire-retarding properties of particleboard of wollastonite nano fibers. The results have showed that fireretarding properties have been improved [19]. Chaiwutthinan et al., have devised a composites of poly (lactic acid)/poly (butylene adipate-co-terephthalate) blend with wood fiber and wollastonite. It is found that elongation-at-break, MFI, thermal stability and impact strength have decreased, but tensile and flexural modulus, tensile and flexural strength have increased [20]. Jose and Elias have devised a novel method to make test specimens from ribbons extruded polybutylene terephthalate/acrylonitrile-butadiene-styrene blends. Extruded samples have better tensile strength, impact, tensile strain at break [21]. Pietro et al., have devised PBT-based nano composites of commercial alumina whiskers to analyze morphological and dielectric properties. The results of the test show the influence of the filler content on various properties like volume electrical resistivity [22]. Ertug has examined probable use of coal bottom ash waste in cement pastes. The devised composite is light in weight which is very ideal for brick, paving stone, tile and applications required low strength [23]. Maharaja et al., have used bottom ash to reinforce Metal Matrix Composites (MMC). The devised composite with varying percentage of bottom ash has improved mechanical properties [24].

The literature review in the area of polymer composites reveals that use of bottom ash as filler material has not been done with Polybutylene Terephthalate (PBT) polymer. In this research we have made PBT composite of bottom ash with and without coupling agent and its mechanical and thermal properties have been found out. Bottom ash filled PBT composites were prepared by adding three different sizes of bottom ash in wt % of 5, 10, 15, 20, 25, and 30 to the polymer without coupling agent. Mechanical properties of the composite were found out which resulted in optimum results for size of 90 microns of the bottom ash and loading of 15% by wt. Coupling agent was added in this polymer composite with concentration of 2%, 4%, 6%, 8% and 10% and the resultant composite was tested for mechanical properties.

## 2.0 MATERIALS AND METHODS

#### 2.1 Materials

Polybutylene Terephthalate (PBT) resin grade (Figure 1) Crastin® S620F20NC010 was obtained from DuPont Petrochemicals Limited Mumbai. This grade is an unreinforced, nucleated, lubricated, medium viscosity for fast injection molding and provides excellent performance in many applications. Bottom Ash was obtained from KALPANA MINERALS PVT. LTD. Sion, Mumbai. The Bottom Ash (Figure 2) was separated in three different particle sizes of 90, 150 and 300 microns using screen sieves. Vinyltrimethoxy silane (VTMO) coupling agent was obtained from Krishna Enterprise, Borivali, Mumbai.



Figure 1 Polybutylene Terephthalate Resin Polymer



Figure 2 Bottom Ash of Thermal Power Plant

## 2.2 Methods Of Preparation Of Test Specimens

## 2.2.1 Dry Blending

Crastin PBT resin and Bottom Ash were preheated in an air circulated oven at the temperature of 105°C for 4 hours. Untreated Bottom Ash (UTBA) was added in different concentration of 5%, 10%, 15%, 20%, 25% and 30% by wt to PBT resin for each particle size (90, 150 and 300 microns) of Bottom Ash. Mixing was done in a tumbler mixer in different proportions of Bottom Ash with Crastin PBT resin for 5 minutes. The 90 microns particle size Bottom Ash was treated with VTMO coupling agent with 2%, 4%, 6%, 8%, 10% by wt concentration of Silane. This treated Bottom Ash (15% by wt) was then added to Polybutylene Terephthalate. Weight % of PBT resin and Bottom Ash of size 90, 150 and 300 microns is given in table 1.

Table 1 Weight % of PBT Resin and Bottom Ash (Filler) of size 90, 150 and 300  $\mu$ 

	•				
S.R.	Batch	Polymer	Filler wt	Filler wt	Filler wt
N.	(PBT resin +	wt	%	%	%
	Bottom	%			
	ash)		90 μ	150 μ	300 μ
1	PBT/UTBA-0	100	0	0	0
2	PBT/UTBA-1	95	5	5	5
3	PBT/UTBA-2	90	10	10	10
4	PBT/UTBA-3	85	15	15	15
5	PBT/UTBA-4	80	20	20	20
6	PBT/UTBA-5	75	25	25	25
7	PBT/UTBA-6	70	30	30	30

## 2.2.2 Melt Compounding

The Melt compounding of bottom ash mixed PBT was done on co-rotating twin screw extruder (Figures 3 and 4) which has screw diameter 16 mm, L/D ratio of 38:1 and screw rpm 60. The temperature for melt compounding of extruder zones is given in table 2. Bottom Ash filled Crastin PBT resin composite (the extruded plastic material) was pelletized on Mayuresh pelletizer. Range of 600-800 rpm was set for pelletizer machine.

Table 2 Extruder zones temperature for melt compounding								
	Zone1	Zone2	Zone3	Zone4	Die			
Temp (°C)	230	240	250	255	260			



Figure 3 Twin Screw Extruder



Figure 4 Cooling Trough

#### 2.2.3 Injection Molding

Family injection mold was used for molding of test specimens required for various mechanical tests. Family mold consisting of cavities for tensile and impact test samples was used to prepare samples for tensile, impact and flexural testing as per ASTM standards. Temperature profile of injection molding machine (Figure 5) is given in table 3.

Table 3 Temperature profile of Injection Molding Machine									
Temp.	Zone 1	Zone 2	Zone 3	Zone 4	Nozzle				
Set value	90°C	220°C	230°C	240°C	245°C				
Actual value	107°C	218°C	230°C	239°C	237°C				



Figure 5 Injection Molding Machine

#### 2.2.4 Mechanical Properties

The tensile strength of specimen (Figure 6) was found according to ASTM D638 using universal testing machine. The cross-head jaw speed of 50 mm/min was maintained for testing and load cell of 0-1 ton was used. The result reported for an average of 5 test specimen.

Rectangular test bar of dimension 120mm x 12mm x 6mm (Length x width x thickness) molded from injection molding. Izod impact strength was measured as per ASTM D256 by using pendulum type impact strength tester, with 2.7 J striker. At least five specimen average value resulted.

Flexural strength of the composite material is measured by standard test method ASTM C78 (third point loading) or ASTM C293 (centre point loading). Flexural strength is expressed as modulus of rupture in MPa.

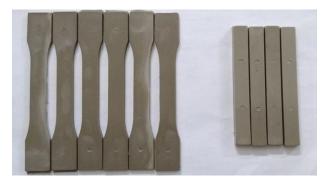


Figure 6 Test Specimen

## 2.2.5 Morphological Properties

The fractured samples from tensile test were characterized using JSM-6380LA analytical scanning microscope of Joel make, Japan. The micrographs of the samples were recorded and studied.

## 3.0 RESULTS AND DISCUSSION

Since composition of 15% Bottom Ash (90  $\mu$ ) and PBT resin polymer gives optimum results, coupling agent was mixed in 2%, 4%, 6%, 8% and 10% (of Bottom Ash) with this composite

polymer and treated Bottom Ash PBT polymer was made and tested for different mechanical properties. The results obtained are tabulated along with mechanical properties of untreated Bottom Ash filled PBT composite.

## 3.1 Tensile Strength

Figure 7 shows that tensile strength of the said composite has been more than that of the virgin PBT polymer. It is also observed that the tensile strength of Untreated Bottom Ash filled PBT composite reduces when the weight percentage of the Untreated Bottom Ash becomes more than 15% due to agglomeration of the bottom ash in the composite. Figure 7 shows that the said PBT composite has more tensile strength for untreated bottom ash size of 90 micron than that of 150 microns and 300 microns as the smaller size of the filler material gives larger surface area for bonding. From the figure 7 and figure 8, it has been observed that there is a marginal improvement in the tensile strength of treated bottom ash filled PBT composite over the Untreated Bottom Ash filled PBT composite.

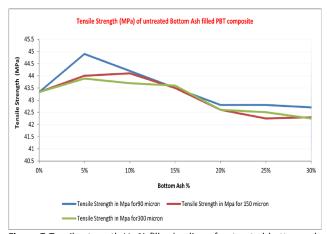


Figure 7 Tensile strength Vs % filler loading of untreated bottom ash filled PBT Composites

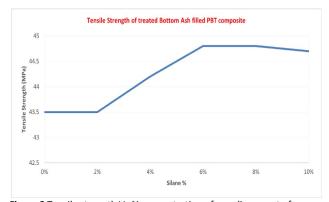
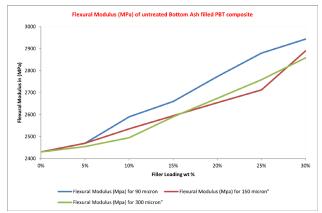


Figure 8 Tensile strength Vs % concentration of coupling agent of treated bottom ash filled PBT Composites

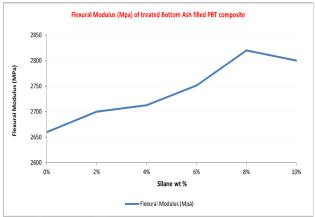
## 3.2 Flexural Modulus And Flexural Strength

Figure 9 shows that flexural modulus of the said composite is significantly increased than that of the plain PBT polymer. From the figure 9 it is observed that the said PBT composite has more flexural modulus for Untreated Bottom Ash size of 90 micron than that of 150 microns and 300 microns as the smaller size of

the filler material gives larger surface area for bonding. From the figures 9 and 10 it has been observed that there is a marginal improvement in the flexural modulus of treated Bottom Ash filled PBT composite over the Untreated Bottom Ash filled PBT composite.

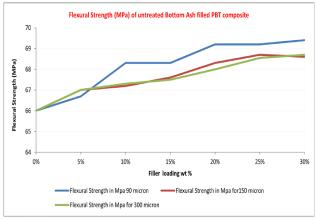


**Figure 9** Flexural modulus Vs % filler loading of untreated bottom ash filled PBT Composites



**Figure 10** Flexural modulus Vs % concentration of coupling agent of treated bottom ash filled PBT Composites

Figure 11 shows that flexural strength of the said composite is increased than that of the virgin PBT polymer. From the Figure 11, it is observed that the said PBT composite has more flexural strength for untreated Bottom Ash size of 90 microns than that of 150 microns and 300 microns as the smaller size of the filler material gives larger surface area for bonding. From the figure 11 and 12, it has been observed that there is a slight improvement in the flexural strength of treated Bottom Ash filled PBT composite over the Untreated Bottom Ash filled PBT composite.



**Figure 11:** Flexural strength Vs % filler loading of untreated bottom ash filled PBT Composites.

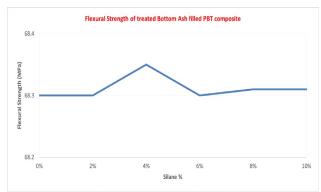
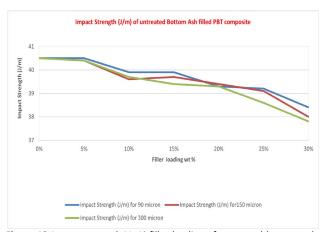


Figure 12 Flexural strength Vs % concentration of coupling agent of treated bottom ash filled PBT Composites

## 3.3 Impact Strength

Figure 13 shows that impact strength of the said composite remains same up to 5% weight of the filler material (90 microns) and thereafter it drops marginally with increased weight % of filler material that of the plain PBT polymer. From the Figure 13, it is observed that the said PBT composite has more impact strength for a filler material (Untreated Bottom Ash) size of 90 micron than that of 150 microns and 300 microns as the smaller size of the filler material gives larger surface area for bonding. From the Figure 13 and Figure 14, it has been observed that the impact strength of both treated Bottom Ash filled PBT composite and the Untreated Bottom Ash filled PBT composite are almost same.



 $\begin{tabular}{ll} \textbf{Figure 13} & \textbf{Impact strength Vs \% filler loading of untreated bottom ash filled PBT Composites. \end{tabular}$ 

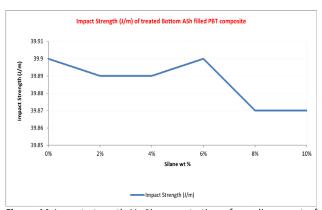
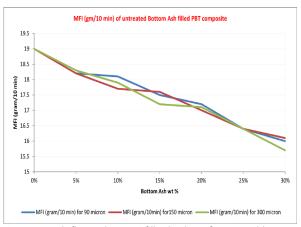


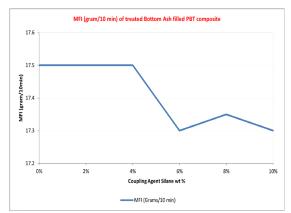
Figure 14 Impact strength Vs % concentration of coupling agent of treated bottom ash filled PBT Composites

## 3.4 Melt Flow Index

Figure 15 shows that Melt Flow Index of the said composite drops marginally than that of the plain PBT polymer. This may be attributed to increase in viscosity due to addition of bottom ash. From the Figure 15 and Figure 16, it has been observed that the Melt Flow Index of both treated Bottom Ash filled PBT composite and the Untreated Bottom Ash filled PBT composite remains same. Thereafter it drops marginally with increased % of coupling agent as compared to that of Untreated Bottom Ash filled PBT composite.



 $\begin{tabular}{ll} \textbf{Figure 15} & \textbf{Melt flow index Vs \% filler loading of untreated bottom ash filled PBT Composites} \end{tabular}$ 



**Figure 16** Melt flow index Vs % concentration of coupling agent of treated bottom ash filled PBT Composites

#### 3.5 Fracture Morphology

Figures 17, 18 and 19 show SEM images of tensile fractured specimens for 15%, 20%, 25% Untreated Bottom Ash filed PBT composites respectively. It is evident from these micrographs that there is a uniform dispersion of Bottom Ash in said composite at 15% loading of treated as well as Untreated Bottom Ash whereas agglomeration is seen at 20% loading of Untreated Bottom Ash and improper bonding is observed for a loading of 25% of Untreated Bottom Ash.

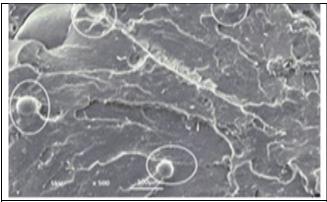


Figure 17 SEM Image of 15% untreated bottom ash filled PBT composite.

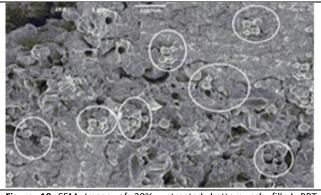


Figure 18 SEM Image of 20% untreated bottom ash filled PBT composite

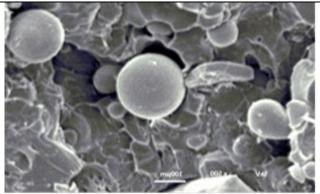


Figure 19: SEM Image of 25% untreated bottom ash filled PBT composite.

#### 4.0 CONCLUSION

Untreated and treated Bottom Ash filled PBT composites show similar tensile and impact properties at 15% loading of the said filler material with 90 microns particle size. However, there is significant improvement in flexural properties of (both treated and untreated) said composite at 15% loading than that of the virgin polymer matrix. During experimentation processing difficulties were encountered with filler loading beyond 15%. Since there is no additional processing cost while preparing this composite, it is concluded that cost of the expensive polymer such as PBT can be reduced by 15% by the addition of 15% of Bottom Ash. It is also concluded that it is an effective and novel way of disposal of Bottom Ash which is an environmental hazard otherwise. This research can be further extended towards replacement of glass fibres in engineering polymers by using Bottom Ash.

## Acknowledgement

I am very grateful to the Institute of Chemical Technology, Mumbai for giving me an opportunity to use its infrastructure for this research work. I am equally thankful to my organization Veermata Jijabai Technological Institute, Matunga Mumbai, for allowing me to carry out the research work

## **Conflicts of Interest**

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper

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