



## Effect of Barium sulphate on Tensile, Flexural and Radiopaque properties of LDPE/BaSO<sub>4</sub> Composites

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**Abstract** —Study related to Low Density Polyethylene (LDPE)/Barium Sulphate (BaSO<sub>4</sub>) composites were done. Barium sulphate (BS) was mixed with LDPE at 0, 10, 20, 30, 40 and 50 wt%. A twin screw extruder was used to mix BaSO<sub>4</sub> with LDPE at 140 °C and granules were prepared. Specimens of the batches thus made were prepared by injection molding. Mechanical properties and radiopacity of the samples were studied and compared and it was found that they increase with the increase in Barium sulphate content.

**Keywords-** Low density polyethylene, Radiopacity, Barium sulfate, X-Ray

### I. INTRODUCTION

Plastics have superior design flexibility compared to metals, ceramics and glass. The products made from plastics can be made into different shapes, sizes, thickness and color to meet the physical, mechanical, chemical and biocompatibility requirements. Plastics can be processed by various processing techniques with the addition of fillers and additives. Radiopacity or clear visibility under X-ray is highly required for plastics used in medical devices. Various Radiopaque additives are added to the plastics for this purpose. Medical devices used for diagnostic and other interventional surgical procedures require clear visibility under X-Ray.

The Radiopaque additives render plastic visible under X-rays and work as well as lead without any of the toxic side effects or disposal concerns. Devices like catheters, guide tubes, surgical tools, dental products, stents and balloons that are used inside the body, in many cases, need to be opaque to fluoroscopy or X-rays so that the surgeons are able to see the devices as it is guided through or placed in the body. Most plastics are transparent to X-rays and require additives that are radiopaque. The most common radiopaque additives are Barium Sulphate, Bismuth compounds, Tungsten and Tantalum.[1]

Barium sulphate (BaSO<sub>4</sub>) is a radiopaque additive. Its specific gravity is 4.5. It is obtained from naturally occurring mineral known as Blanc Fixe having an average particle size less than 2 micrometers and is found in varying purities around the world. Deposits are commercially mined in the U.S. and Canada and are one of the densest minerals in use. [2] It is a white powder used as filler because of its unique characteristics like X-Ray opacity, specific gravity, frictional resistance, chemical resistance. [3]

Almost all thermoplastics like commodity, engineering and high performance plastics are widely used in medical device applications. LDPE is a thermoplastic material which is a material of choice for medical applications like packaging films, tubings, and IV components to hip and joint replacement implants. It has lower tensile strength and increased ductility. The high degree of branching with long chains gives molten LDPE unique and desirable flow properties.

#### 1.1 Literature Survey

Quin Peng [4] studied radiopaque materials containing bismuth oxychloride. Bismuth oxychloride is an unique filler and renders many advantages over other fillers for radiopaque medical devices. It provides high radiopacity and sharper contrast image than barium sulfate, allows high loading without adverse effect on the mechanical properties of the polymers and facilitates more efficient manufacturing process because of its excellent powder dryness. Furthermore, bismuth oxychloride can improve the laser mark property of the plastics. Guangyu Lu (2004) et.al [5] studied the Rheology and Processing of BaSO<sub>4</sub> filled Medical- Grade Thermoplastic Polyurethane. It was observed that 20% vol of BaSO<sub>4</sub> filled TPU exhibits decreased rather than increased, shear viscosity and elasticity in comparison to unfilled TPU at 190 to 200 degree centigrade at which these materials are usually processed. Ke Wang (2003) et.al [6] studied the effect of mechanical properties and toughening mechanisms of polypropylene/barium sulfate composites. It was found that PP can be toughened with specially treated BaSO<sub>4</sub> particles. Nirmala James (2005) et.al [7] studied Polyurethanes with radiopaque properties. An aliphatic, commercially available, medical grade polyurethane, Tecoflex 80 A was made radiopaque by coupling a 5-iodine-containing molecule.. Ke Wang (2005) et.al [8] studied the microstructure and fracture behavior of PP/ BaSO<sub>4</sub> composites. Four composite samples with different PP- BaSO<sub>4</sub> interface were prepared by treating the filler with different modifiers. The fracture behavior of the composite samples under different strain rates was studied. Mario Tanomaru-Filho (2007) et.al [9] studied the radiopacity Evaluation of New Root Canal filling Materials by Digitalization of Images. The authors concluded that reduction in the amount of radiopaque substances in the sealers

led to a decrease in radiopacity. N.W.Elshereksi (2009) et.al [10] studied effect of filler incorporation on the fracture toughness properties of Denture Base Poly (Methyl Methacrylate). The aim of the study was to investigate the possibility of using barium titanate as a radiopacifier in PMMA. Siti Shuhadah\* and A.G. Supri (2009)[11] studied the effects of chemical modification on the mechanical properties, morphology and water absorption of low density polyethylene/egg shell powder composites. Both the introduction of interfacial adhesion to composites and better interaction adhesion between LDPE and egg shell powder are responsible for the improvement of the mechanical properties of the LDPE/ESPI. Rohani Abdul Majid (2010) et. al[12] studied effects of polyethylene-g-maleic anhydride (PE-g-MA) as a compatibilizer on tensile properties and morphology of low density polyethylene/thermoplastic sago starch (TPSS)-kenaf fibre composites. Results indicated that the tensile strength and Young's modulus of LDPE/TPSS-kenaf fibre composites with the addition of PE-g-MA were greater than the composites without the addition of PE-g-MA particularly at higher fibre loading. S. Kiran (2012) et.al [13] studied Synthesis and characterization of three different radiopaque thermoplastic polyurethane elastomers.. The polyurethanes developed have radiopacity equivalent to radiopacity of 20 wt % BaSO<sub>4</sub> filled polymers. Shouhai Li (2013) et. al [14]studied the effects of compatibilizers on composites of Acorn shell powder and LDPE . The mechanical properties of composites decreased with increasing acorn shell content. Salma Zamirian (2013) [15] studied a comparison on Mechanical Properties on LDPE/Clay and LDPE-g-MA/Clay Nanocomposites for Film Packaging Applications Samples were prepared with the nano content 1 to 5 wt % and their mechanical properties such as Tensile Strength, Young's modulus, Impact strength have been considered. Vincent L.Vaillancourt (1970) [16] invented X-Ray opaque catheter tubing for intravenous use or analogous uses which is composed of polytetrafluoroethylene or polyfluorinated ethylene-propylene("Teflon," "TFE" or"FEP") with a suitable filler .Edmund H.Carus and Eric F. T. White (1985) [17] invented a radiopaque fiber comprising polypropylene, a coupling agent and 55% to 70% of barium sulfate. The fiber finds particular application in manufacture of surgical swabs and dressings.Michael R Lange et.al.(1999) [18] invented radiopaque catheter and method of manufacturing By selecting a high concentration of a radiopaque filler material for an inner layer and a lower concentration of radiopaque filler that is selected to provide a relatively smooth surface, the X-ray visibility of the catheter may be maximized while still maintaining a relative smooth outer surface.

## **II. MATERIALS & METHODOLOGY**

### **2.1 Materials**

The thermoplastic polymer used for making composite was Low density polyethylene manufactured by Reliance industries Ltd and is FDA approved (LDPE 24 FS040 grade). Its MFI is 4gm/10min. Barium sulfate (BS) used is Blance Fixe Super F grade is FDA approved manufactured by Sachtleben Chemie GmbH, Germany and supplied by M/s. Chemtex Materials Pvt. Ltd.Odhav, Ahmedabad. The BaSO<sub>4</sub> is 99%, average particle diameter is approx 1µm, the hardness is 3(Mohs),is non-toxic and is free of impurities such as quartz. The dispersibility, weather resistance and gloss is excellent.

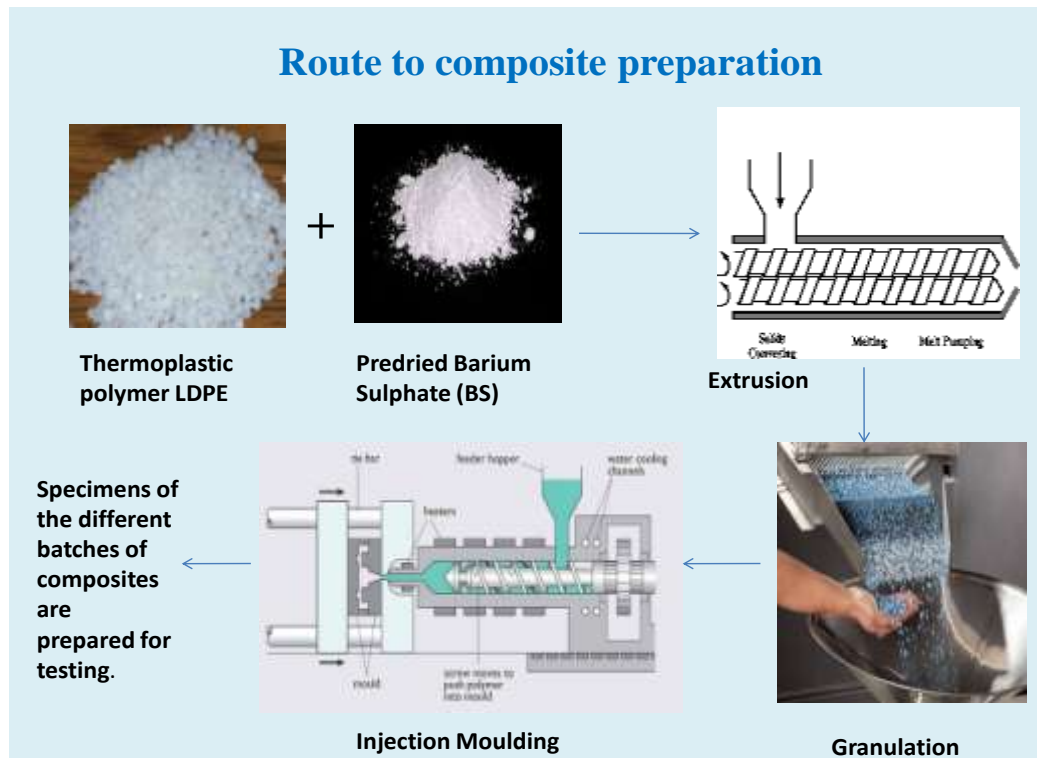
### **2.2 Composite Preparation**

LDPE/Barium sulfate (LDPE/BS) composites were prepared by using Co-rotating Twin Screw Extruder with sections for feed, high shear, and high intensity mixing and discharge on a continuous basis. The screw diameter was 21 mm, L/D ratio of the screw was 40:1 and motor rpm was kept 258. The melt temperatures were kept 140-150 °C. Batches of 3 kg each were prepared with 0, 10, 20, 30, 40 and 50 wt % loading of BS into LDPE. The various batch compositions for preparing composites are given in Table I.

**Table I: Composition of LDPE and Barium Sulphate**

<b>BATCH ( 3 Kg )</b>	<b>COMPOSITION</b>
LDBS0	LDPE + BS 0 wt%
LDBS10	LDPE + BS 10 wt%
LDBS20	LDPE + BS 20 wt%
LDBS30	LDPE + BS 30wt%
LDBS40	LDPE + BS 40 wt%
LDBS50	LDPE + BS 50 wt%

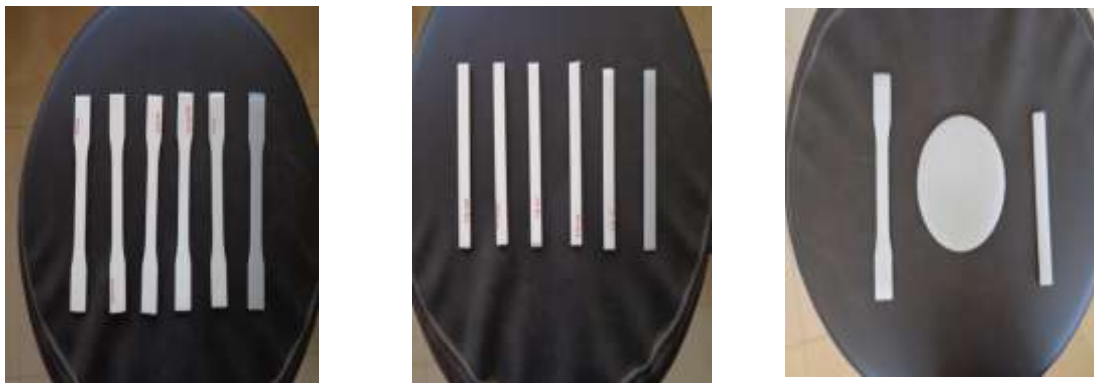
The method of preparing composites is shown in Figure I



**Figure 1: Method of Composite Preparation**

### 2.3 Sample Preparation

The test specimens for various tests were prepared by using Automatic Injection Molding Machine. Before loading the material in the hopper the composites was predried at 65 °C to remove moisture and allowed to cool at room temperature for after removing from the hopper. The injection molding was carried out at 160-170°C and different test specimens of dumbbell bar and disc shapes were prepared to carry out various tests. Figure 2 shows the samples of composites prepared by Injection molding.



**Figure 2: Various Specimens Prepared From Granules Of LDPE/BS Composites**

### 2.4 Characterization of Composites

Tensile strength by ASTM D 638 method and Flexural strength by ASTM D 790 method were measured .X-ray analysis was done through a digital X-ray machine manufactured by AGFA Company Model No CR 35-X. The specimens of different composites were tested from a distance of 88cm at 46 KV and 4 MAS radiation and the behaviour of the composites to X-rays was observed.

### III.RESULT AND DISCUSSION

#### 3.1 Tensile Strength and Elongation

Figures 3 and 4 show the effect of BS content on tensile strength and elongation on LDPE composites

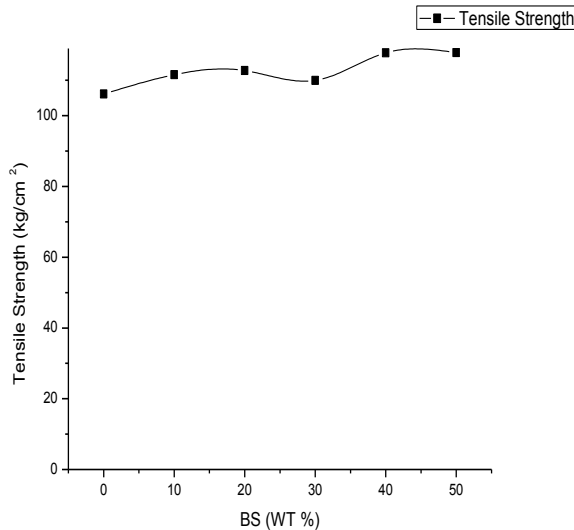


Figure 3: Effect of BS content on tensile strength

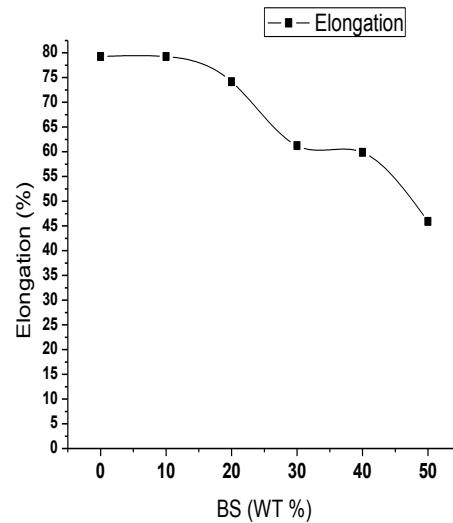


Figure 4: Effect of BS content on elongation

Fillers affect tensile properties according to their packing characteristics and interfacial bonding. Tensile Strength increases with the increase in the BS content. This is because of good dispersion of the filler in the matrix and good interfacial bonding between the matrix and the filler[11]. Good dispersion of the filler is contributed due to 1 micron particle size. The % elongation decreases with the increase in the BS content [5]. This might be due to the reduction in toughness of the composite.

#### 3.2 Flexural Properties

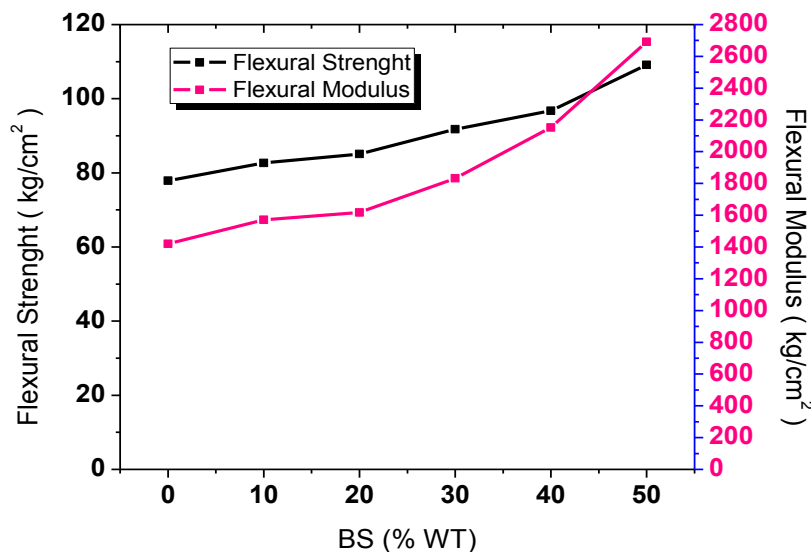


Figure 5 : Effect of BS content on Flexural strength and modulus

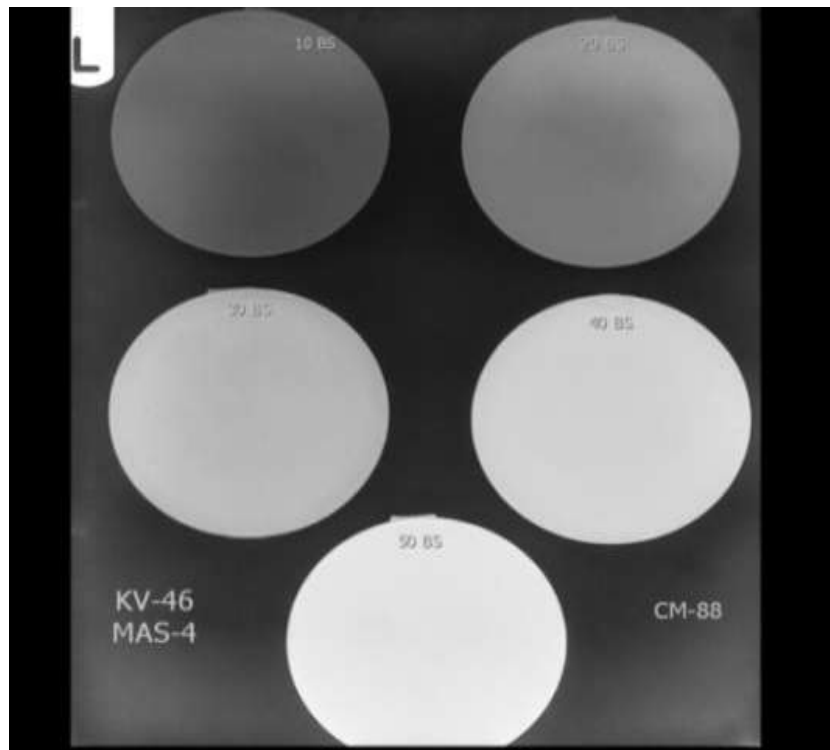
Figure 5 shows that flexural strength and modulus increases with the increase in the BS content. This is due to increase in stiffness of the composite because of good dispersion and interfacial bonding of the filler in the polymer matrix.[11] Table 2 shows the summary of Mechanical Properties of LDPE/BS Composites.

**Table 2 : Summary Of Mechanical Properties Of LDPE/BS Composites**

COMPOSITION	Barium Sulphate %	Flexural Strength Kg/cm <sup>2</sup>	Flexural Modulus Kg/cm <sup>2</sup>	Tensile Strength (Kg/cm <sup>2</sup> )	% Elongation
LDBS0	0	77.3914	1419.52	106.12	79.16
LDBS10	10	82.6323	1571.06	111.62	79.25
LDBS20	20	85.1037	1617.30	112.73	74.17
LDBS30	30	91.7441	1832.56	105.95	61.23
LDBS40	40	96.7169	2151.82	117.78	59.88
LDBS50	50	109.134	2692.19	117.80	45.93

### 3.3 X-RAY Test

Figure 6 shows combined X-ray image of all the LDPE/BS composites. It can be seen that X-ray opacity decreases with the decrease in BS content and increases with the increase in BS content. Barium Sulphate makes the composites radiopaque [18]. This is because of its high density and relatively high atomic number ( $Z = 56$ ) of Barium, due to which it absorb X-rays more strongly.



**Figure 6: Combined X-Ray images of all the LDPE/BS composites**

## IV. CONCLUSION

The effect of radiopaque additives on mechanical properties and radiopacity were studied by conducting Tensile, Flexural and X-Ray tests on the samples. The Tensile strength, Flexural Strength and Radiopacity increases with the increase in BS content in composite whereas elongation decreases. Good dispersion of BS particles in the LDPE matrix results in good mechanical properties of the composites. LDPE/ BS composites thus prepared can be used in applications like medical devices where X-Ray Opacity is of prime importance. The composites prepared will have a significant good mechanical strength and radiopacity and it will create scope for further research in this area.

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