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Experimental Investigation of Mechanical Properties of Impact Modified Polyvinyl Chloride-Fly Ash Composites

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Abstract

The mechanical properties like tensile, pin bearing and impact properties have been studied for composites containing Fly Ash (FA) and Impact modifier (IM) in Polyvinyl Chloride (PVC) for furniture and pipe applications. Pin-bearing test is an important tool to evaluate the ability of the material to retain fasteners and the ability of the material to sustain load during the service life. The present paper evaluates the effect of variation of FA and IM on the pin bearing strength, tensile strength, modulus elongation at break and impact strength. Scanning electron microtopography indicates that the impact modifier forms a co-continuous phase at 10% of IM in PVC. Increase in void content decreases impact strength and tensile strength. As void content increases moisture absorption also increases. The results of pin bearing tests were analogous to tensile test and correlate well. Results were in accordance with impact modification theory. Moisture absorption was studied keeping in view outdoor applications.

Keywords

Flyash-PVC Composites, Impact Modifier, Pin Bearing Properties, Tensile Strength

1. Introduction

Impact modified and filled grades of PVC are used mainly for their inherent high stiffness to cost ratio [1]-[9]. Fillers are mainly used in the present day formulation of PVC for reducing cost, the major filler being calcium carbonate. Needless to say, fillers like calcium carbonate, talc and FA may or may not act as reinforcing fillers. Cost reduction is at the expense of desired properties. They are seen to affect certain properties like pin bearing strength and impact strength [10]. Impact modifier is added to improve the impact strength [11]. FA is emerging as the major filler in polymeric systems [12] [13] [14] [15] [16]. FA is the by-product of burning coal in power plants. Disposal and use of FA is a major issue and it is a potential contaminant for surface and ground water. FA has been used as filler in various resins like HDPE, LDPE and PP [17]. Khoshnoud *et al.* studied the mechanical properties of in PVC. Addition of fly ash showed enhancement in mechanical properties [18]. Coupling agents are used for improving mechanical properties. It was observed that addition of silane coupling agents enhanced the mechanical properties of PP-FA composites [16]. Deepthi *et al.* used FA cenospheres as fillers in HDPE matrix. The results showed that surface modification of Cenospheres accompanied by compatibilization led to the substantial improvement to mechanical properties and thermal stability of the composites [19]. Feasibility tests of using flyash samples as filler in PVC indicated that sodium lauryl sulphate treated fly ash could successfully replace $CaCO_3$ as filler in PVC under conditions of low filler loading [20]. Khoshnoud *et al.* studied the mechanical, thermal and morphological properties of PVC foams filled with flyash, glass fibres and mica. They observed increase in mechanical properties with addition of all the three fillers [18]. PVC foam-fly ash composites were characterized for their structural, morphological, mechanical and thermal properties by Khoshnoud *et al.* The tensile strength of the composites was seen to increase modestly with higher fly ash loading, while there was a significant increase in the elastic modulus for the same composites [18]. Rigid Polyvinyl Chloride (PVC) foam composites are one of the most common materials used in building industry in the form of profiles, sheets, and pipes due to their low cost, low density, low thermal conductivity, improved acoustic damping properties, and good fire retardancy [21]. Kabir *et al.* proposed a general model comparison of tensile properties of HDPE-filled fly ash composites. The mechanical properties were seen to depend on the shape, size and volume of fly ash [22]. Wang *et al.* studied the rheological behavior of plerospheres filled UPVC using Brabender plasticorder. Plerospheres are defined as superfine spherical particles (0.5 - 5 microns) separated from FA. It was observed that lower torque was required with silane-coated plerospheres than uncoated plerospheres [23]. Effect of FA on properties of nylon 6 revealed that the mechanical property of composite is a function of the particle size, the dispersion, the particle orientation, the interfacial interaction between the minerals and the polymer matrix [24]. The effect of bis-(3-triethoxysilylpropyl)-tetrasulfide, Si69 on the tensile properties, swelling behavior and morphological characteristics of recycled poly(vinyl chloride)/acrylonitrile–butadiene rubber/fly ash (PVCr/NBR/FA) composites was studied by Ismail *et al.* It was observed that Si69 increased the interfacial interaction between the matrix and FA leading to enhanced mechanical properties [25]. Study of mechanical properties of PEEK-FA composites showed enhancement in mechanical properties on using a compatibilizer [26].

Addition of filler of any form—particulate, fibrous, flasks etc. into the polymer matrix reduces the ductile behavior leading to poor impact strength. Impact strength can be enhanced by addition of impact modifiers. The improvement in impact strength obtained depends on type and proportion of the modifier and processing conditions. For a thermoplastic to be rendered impact resistant, a multiphase structure containing a "hard" thermoplastic phase and a "soft" elastomeric phase is essential. This arises due to the incompatibility between the thermoplastic and the impact modifier, which is an elastomer [27].

Impact modifiers like EVA and chlorinated polyethylene have elastomeric phase distributed as a honeycombed network whereas with Methymethacrylate Butadiene Styrene (MBS) and all acrylic impact modifiers spherical elastomeric particles are dispersed in the hard phase. In the later case, the spherical particles retain their shape and lesser shear sensitiveness gives wide processing range. All acrylic impact modifiers are manufactured by graft copolymerization of acrylic monomers like butyl acrylate with methyl methacrylate. The process used is emulsion polymerization. In the first step a core of polybutyl acrylate is polymerized and subsequently a shell of poly methyl methacrylate is grafted on. This hard shell improves their anchoring in PVC matrix. The shell—core morphology forms the secondary particles, which break down to primary particles of size 0.2 μm on processing. These primary particles absorb impact energy and, thereby, prevent brittle fracture [28] [29].

Apart from various other applications of un-plasticized PVC, major application of it is in the pipes and fittings. Pipes and fittings require impact properties, good tensile strength, short term and long-term pressure sustainability (ASTM D 1785). Fittings also require good pin bearing properties as they are threaded, and screwed. Though tensile and impact properties of PVC-FA have been studied by many researchers, there is no precedence of study of pin bearing properties of PVC-FA composites. Present paper evaluates the effect of fly ash (FA) as filler and impact modifier (IM) on un-plasticised PVC. The work presented here is organized as follows. Experimental work is described in Section II, results of the work are discussed in Section III. Section IV summarizes the conclusions of this work.

2. Materials and Method

Suspension PVC grade 57 GE R01 manufactured by M/s Reliance India limited, India was used. FA of Class F as per *ASTM C* 618 with the trade name Pozzoplast, procured from DIRK India Pvt. Ltd., Eklahare, Nasik, India, was used as the filler. Pozzoplast is a specialized FA, resulting from the combustion of pulverized bitumous coal. FA contains SiO_2, MgO, Al_2O_3, Fe_2O_3, Na_2O, Reactive Silica and Chlorides [30]. The particulate size analysis for PVC and FA was done using *Model* 780 *AccuSizer* of Particle Sizing Systems, Inc., USA. The Fly ash has the smallest size but is comparable to PVC. Fly ash with number weight mean diameter of 2.64 μm and standard deviation of 3.16 μm was used. FA used in this experimentation contains 0.5% organic matter which was determined by finding out loss in weight on heating at 700˚C for two hours. PVC with number weight mean diameter of 3.59 µm and standard deviation of 11.32 µm was used. All acrylic IM grade *Paraloid KM*-355*P* made by Rohm and Haas Plastics Additive was used. Sample Coding systemis based on % of PVC. The numbers stand for as a % of PVC of the constituent (**Table 1**).

Processing was carried out in three steps. In the first step, PVC was formulated in a high speed mixer. Additives like lubricant (paraffin wax, calcium stearate and stearic acid), heat stabilizers (tribasic lead sulphate), and acrylic based processing aid were added in first stage. Impact modifier was added in the second stage. FA was added at the final stage. Further compounding was carried out using two-roll mill and the hide was reduced in size using a scrap granulator. The granulated material was extruded in the form of sheet using counter rotating twin-screw extruder attachment of *Plasticorder Brabender, model number AEV* 651. Type IV samples as per ASTM D-638 were cut from the extruded sheets. The samples were tested on Instron 4204 machine with a 10 KN load cell and strain rate of 5 mm/min. Pin-bearing strength is obtained by tension testing a pin-loaded hole in a flat specimen (**Figure 1(a)** and **Figure 1(b)**). Samples were tested on an Instron 4204 machine with strain rate of 5 mm/min. Bearing strength was calculated as per ASTM D 953. The test was carried out by using standard test specimen with eccentricity to diameter (e/d) ratio equal to two. The failure modes were analyzed with visual inspection. The notched Izod Impact test as per ASTM D256, test method A, was carried out using the ZWICK 5102 impact-testing machine. The Izod specimens were cut using profile cutting machine with high speed milling cutter to avoid the micro cracks. Moisture absorption was carried out as per ASTM D 570. Void content was determined from

Table 1. Formulations.

Sr. No.	Formulation Code	Constituents as % of Total Compound			
		PVC	FA	IM	Additives
1	PVC	89.9	0	0	10.1
2	20 FA	76.2	15.2	0	8.6
3	40 FA	66.1	26.5	0	7.4
4	60 FA	58.4	35.1	0	6.5
5	10 IM	82.5	0	8.3	9.2
6	20 IM	76.2	0	15.3	8.5
7	20 FA + 10 IM	70.8	14.1	7.1	8
8	20 FA + 20 IM	66.2	13.2	13.2	7.4
9	40 FA + 10 IM	62	24.8	6.2	7
10	40 FA + 20 IM	58.4	23.4	11.7	6.6
11	60 FA + 10 IM	55.2	33.1	5.5	6.1
12	60 FA + 20 IM	52.3	31.3	10.5	5.9

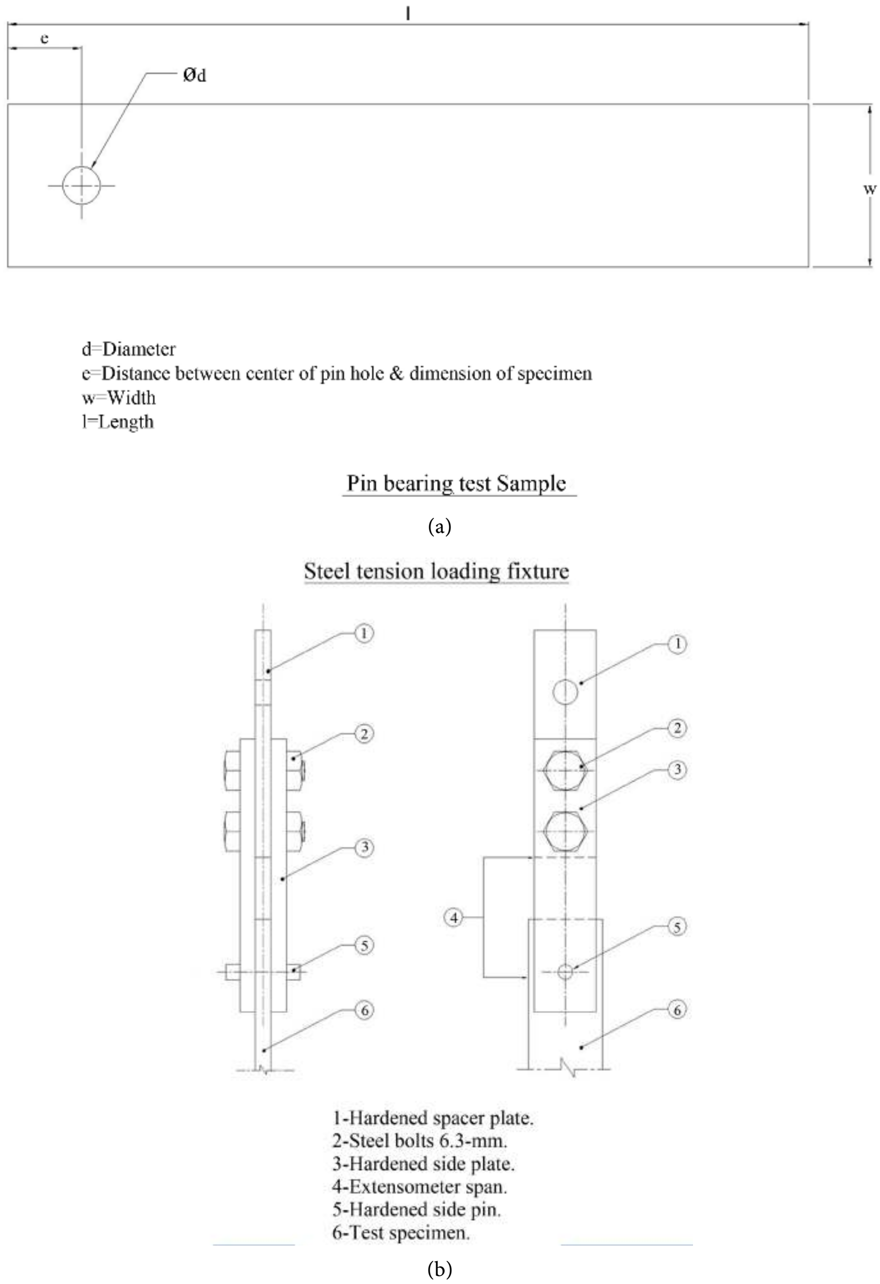


Figure 1. (a) Pin bearing test sample; (b) Pin bearing test fixture.

the theoretical density obtained by rule of mixtures and the experimentally obtained density by Archimedes's Principle. For assessing the morphology of the composites, SEM of representative composites was carried out using *JOEL model JSM* 6360-*A* Analytical SEM. Infrared spectroscopy (IR) of representative samples was carried out using *SHIMADZU* 470 spectrometer.

3. Results and Discussions

3.1. Tensile and Pin Bearing

The results of tensile, pin bearing and impact properties will be considered in tandem as they are analogous to each other. In order to understand the effect of FA and impact modifier separately and in combination, the formulations have been categorized in 5 groups as seen in Table 2. Ultimate tensile strength decreases but Young's modulus increases on addition of FA to PVC as expected (Table 2, Group A). Maximum bearing strength initially increases on addition of 20% FA but later decreases. Bearing strength is defined as strength at 4% hole deformation (ASTM D 953). It is analogous to modulus. The bearing strength increases with increase in FA except 20% FA. Addition of filler induces inhomogenity in the matrix. In absence of a coupling agent, stress transfer between the filler and matrix is poor leading to decrease in the tensile and pin bearing strength. Increase in Young's modulus is due to chain stiffening on addition of filler (Figure 2; Figure 3).

Addition of impact modifier decreases the tensile strength and modulus significantly (Table 2, Group B). Maximum bearing strength and bearing strength also reduces to a substantial extent. Addition of impact modifier increases the flexibility of the matrix leading to decrease in strength and modulus (Figure 4; Figure 5).

Now we discuss the results of Group C, D and E. Addition of FA to 10% and 20% impact modified PVC decreases the tensile strength. Modulus increases in case of addition of 20% FA to 10%IM but remains almost constant on addition of 20% FA to 20IM. Similar trend is observed on addition of 40% and 60% FA to 10% and 20% IM. Bearing strength increases on addition of FA to 10% and 20% IM. This result is in accordance with the results for Young's modulus. There is no significant difference in maximum bearing strength on addition of FA to 10% and 20% IM.

Table 2. Tensile, pin bearing and impact results.

Group	Formulation Code	Moisture Absorption	Void Content	Ultimate Breaking Strength	Young's Modulus	Bearing Strength at 4% Hole Deformation	Maximum Bearing Strength	Impact Strength	Type of Failure
		%	%	MPa	MPa	MPa	MPa	J/m	
	PVC	0.11	2.75	38	2820	14	76	31.3	Bearing to shear out
A	20 FA	0.04	1.11	33	3547	11	84	44.6	Cleavage
	40 FA	0.26	1.97	27	3622	17	63	33.3	Cleavage
	60 FA	0.26	2.67	23	3715	18	63	34.1	Cleavage
B	10 IM	0.03	0.22	28	1536	7	57	95.9	Bearing to shear out
	20 IM	0.6	0.38	15	1045	5	52	55.9	Cleavage
C	20FA + 10 IM	0.25	0.68	18	1903	11	62	59	Shear out to cleavage
	20 FA + 20 IM	0.27	0.83	9	1032	6	43	40.4	cleavage
D	40 FA +10 IM	0.12	0.47	14	1458	10	58	50.1	Shear out to cleavage
	40 FA + 20 IM	0.29	1.08	13	1051	10	51	82.3	Shear out to cleavage
E	60 FA + 10 IM	0.23	0.37	12	1938	13	53	38.4	cleavage
	60 FA + 20 IM	0.30	1.24	9	1131	8	31	77.4	Shear out to cleavage

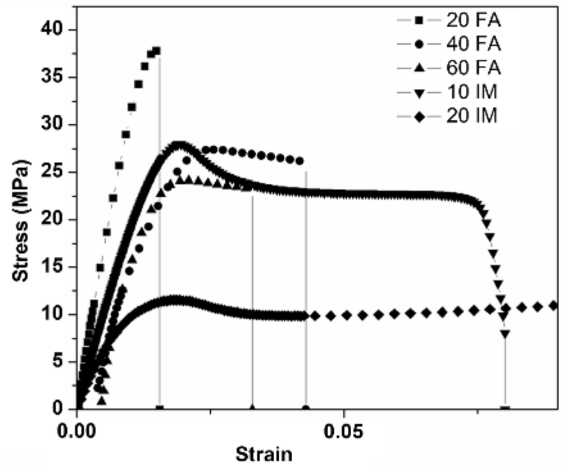


Figure 2. Tensile strength for composites containing 20 FA, 40 FA, 60 FA, 10 IM and 20 IM.

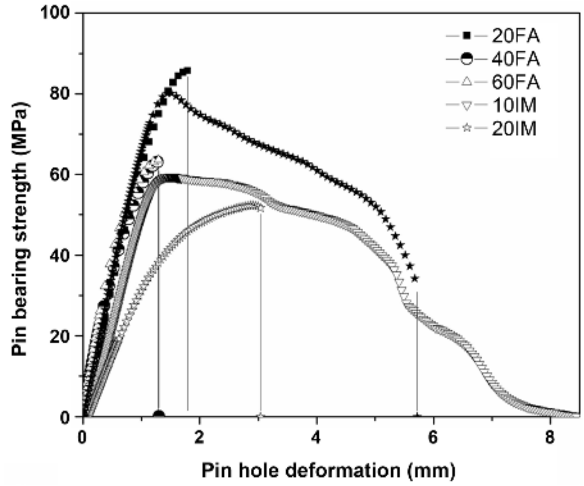


Figure 3. Pin bearing strength at 4% pin hole deformation for 20 FA, 40 FA, 60 FA, 10 IM and 20 IM.

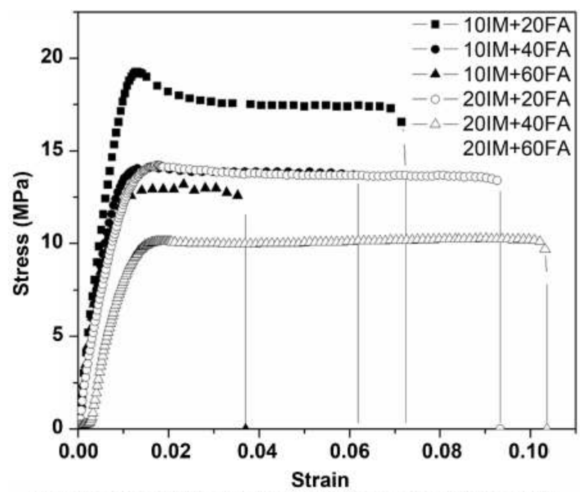


Figure 4. Tensile strength for composites containing 10 IM + 20 FA, 10 IM + 40 FA, 10 IM + 60 FA, 20 IM + 20 FA, 20 IM + 40 FA, and 20 IM + 60 FA.

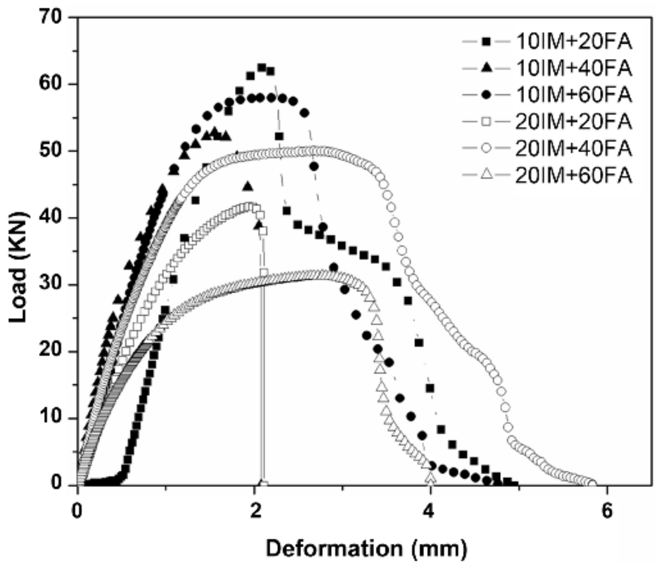


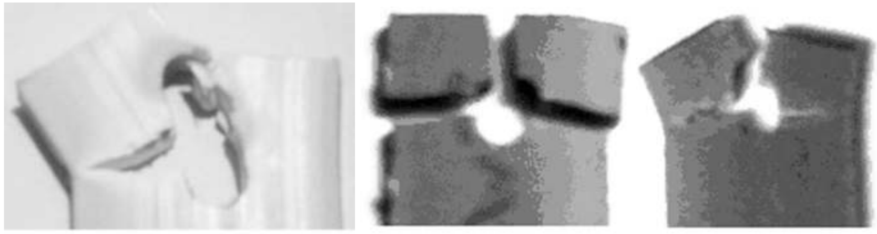
Figure 5. Pin bearing strength at 4% pin hole deformation 10 IM + 20 FA, 10 IM + 40 FA, 10 IM + 60 FA, 20 IM + 20 FA, 20 IM + 40 FA, and 20 IM + 60 FA.

Mode of failure for pin bearing tests depends on the geometry and inherent material properties [28] [31]. The geometry for all the samples for the experimental work in consideration has not been varied. Therefore, the type of failure is only dependent on the inherent material property. Failure may start with any mode but finally leads to ultimate failure by tensile mode. Pure tensile mode of failure is an indication of brittleness due to filler and pure bearing failure is an indication of elastic nature due to impact modifier. Cleavage failure is indicator of orthotropic nature of material due to the possible filler orientation in the extrusion direction. In short, Material becomes more orthotropic in nature leading to predominantly cleavage failure.

PVC shows bearing to shear out failure (**Figure 6(a)**). As there is no filler present in this formulation, there is no chain stiffening and the chains can take up all possible configurations. The tensile modulus of PVC is low. Due to this there is greater deformation of the pin hole before complete failure.

With addition of FA, the mode of failure changes from bearing failure observed in PVC to cleavage failure (**Figure 6(b)**). On further addition of FA, there is an increase in the bearing strength. Maximum strength is found to increase on addition of 20% FA due to reduction is void content. However, void content increases with further addition of FA and strength decreases (**Table 2**, Group A). With addition of IM to PVC, there is a decrease in the bearing and maximum strength and at low content of IM failure is bearing to shear out like PVC but with higher impact modifier the failure mode becomes cleavage indicating orthotropy (**Table 2**, Group B; **Figure 6(c)**).

Addition of IM to composites containing PVC and FA, mode of failure is cleavage or shear out to cleavage. This is an indication of elasticity due to IM as well as orthotropy in the direction of extrusion.



(a) (b) (c)

Figure 6. Bearing failures (a) PVC—Bearing to shear out failure; (b) 20 FA + 20 IM—Cleavage failure; (c) 20 IM + 40 FA—Shear out to cleavage.

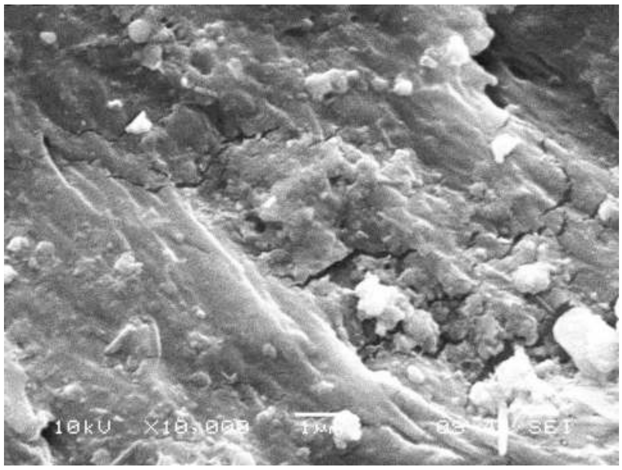
3.2. Impact Strength

The impact strength of 20 FA is more than that of the PVC. This is because FA with its lower particle size occupies the interstitials between PVC particles. This leads to decrease in void content, which results in increase in impact strength. On further addition of FA there is insufficient wetting of FA by PVC. This leads to increase in void content and therefore impact strength remains almost constant on further addition of FA (Table 2, Group A).

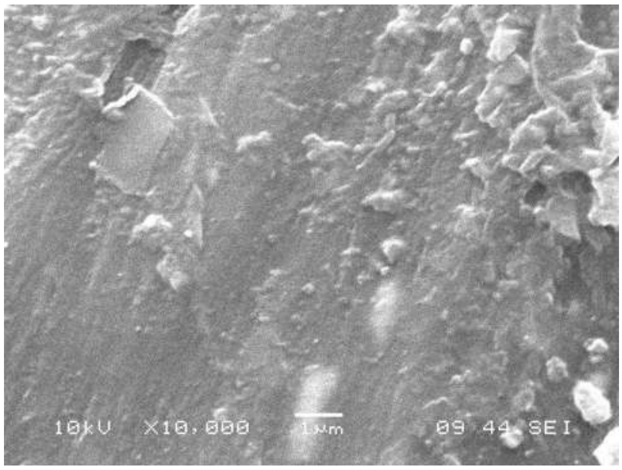
For 10 IM, there is a significant increase in impact strength compared to PVC which is in accordance with Impact Modification theory. In fact highest impact strength is seen here (Table 2, Group B). Further addition of IM *i.e.* 20%, substantial decrease in impact strength is observed which can be attributed to poor dispersion of IM (Table 2, Group B). Addition of 10% IM to 20 FA increases the impact strength initially. This is in accordance with the impact modification theory. There is a decrease in void content on addition of IM, which also contributes to increase in impact strength. On 20% addition of IM, decrease in the impact strength is observed, which can be attributed to poor dispersion of IM (Table 2, Group C). The impact strength of 40 FA + 10 IM is more than 40 FA. There is decrease in void content on addition of IM, which also contributes to increase in impact strength. Further increase in impact strength is observed for 40 FA + 20 IM (Table 2, Groups A and D) Compared to 60 FA, increase in impact strength is observed for 60 FA + 10 IM. This is in accordance with the impact modification theory. There is a decrease in void content on addition of IM, which also contributes to increase in impact strength. Following the same logic still higher impact strength is observed in 60 FA + 20 IM (Table 2, Group E). From this we can conclude that up to certain % IM is properly dispersed in matrix which helps in increasing impact strength beyond optimum amount of IM, there is poor dispersion of the IM leading to lowering of the impact strength.

3.3. Scanning Electron Microscopy and Infrared Spectroscopy

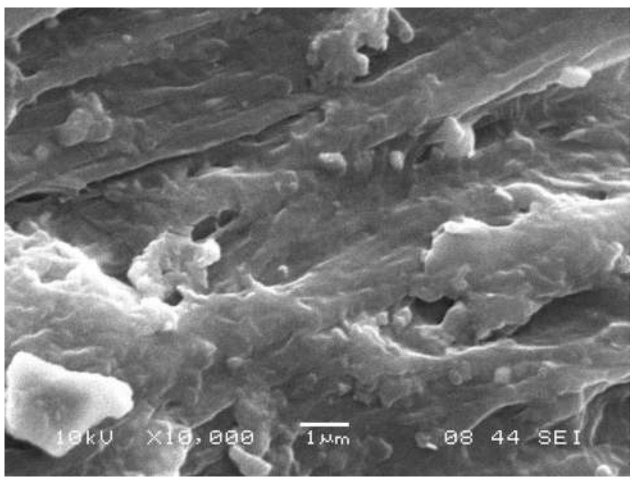
Results of Scanning Electron Micrograph (SEM) support those of the mechanical properties mentioned above. SEM shows that the impact modifier forms a co-continuous phase at 10% IM in PVC (Figure 7(a)). This result is in agreement with increase in impact strength and decrease in tensile and pin bearing strength. SEM of PVC and 20% FA shows FA finely dispersed (Figure 7(b)).



(a)



(b)



(c)

Figure 7. SEM for (a) 10 IM; (b) 20 FA; (c) 10 IM + 20 FA.

This results in decrease in void content and increase in maximum bearing strength and impact strength. In general for the combination of FA and IM, at higher loading dispersion is poor (**Figure 7(c)**).

Except broadening of peak at 1750 cm^{-1} indicative of interaction between organic matter in FA and formulated PVC, no major interaction between the constituents was found from infrared spectroscopy [31].

3.4. Moisture Absorption and Void Content

The small particulate size of the FA is mainly responsible for reducing void content of the composite initially and the void content of the composite increases with further increase in the FA percentage. The percentage of the FA up to which reduction in void content of the composite is possible is function of weight percentages of PVC and varies from one formulation composition to other. FA has lower particle size than PVC and therefore higher surface area. On addition of 20% FA, the FA occupies the interstices. Addition of 20% FA reduces void content therefore reduces moisture uptake. On further addition of FA, wettability decreases due to higher surface area of FA compared to PVC and hence void content increases which will further increase the moisture absorption (**Table 2**, Group A).

The IM also helps to reduce the void content of the composite. Addition of IM required higher energy of processing. Higher energy increases the viscous heat dissipation. Hence processing temperature is required to be kept low. This in turn means that the extrusion takes place at higher die head pressure. Higher die head or higher compaction force leads to reduction in void content. All acrylic IMs consist of hard shell of poly methyl methacrylate (PMMA) enclosing butyl acrylate. PMMA contains C=O group which increases the hygroscopic nature of composites. At 10% of IM, void content observed is less. Therefore, despite presence of IM, moisture/water absorption observed is less. But as IM % increases the effect of IM becomes more pronounced and the % moisture absorptions increases (**Table 2**, Group B). As % of IM in each group increases, % moisture absorption increases. Addition of IM to the composites containing PVC and FA, increases void content with increase in % of IM (**Table 2**, Groups C, D and E).

4. Conclusion

Tensile and bearing properties drop due to addition of impact modifier and fly ash. However, optimum combination of proportions can be worked out. Without compromising much on impact strength, fly ash alone can be effective. Without compromising much on tensile and bearing properties, impact modifier alone can be effectively used. However, when fly ash and impact modifier are both added in combination, drastic reduction in tensile properties is seen (**Table 2**, Groups C, D and E). Mode of failure is found to change depending on the composition and weight % of constituent. Pin bearing properties reduce in proportion to loading of total of fly ash and impact modifier. The results indicate a significant effect of void content which is mainly due to dispersion and die head pressure during processing. It is, therefore, essential to study further the effect of particle size of FA and die head pressure during processing.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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