
Enhancement of High-Density Polyethylene Properties by Impregnation with Inorganic Alumina Filler

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Abstract: The influence of filler size and content on the mechanical and rheological properties (thermal conductivity, impact strength, hardness and melt flow index (MFI) of Al₂O₃/high-density polyethylene (HDPE) composites have been studied. Concentration of alumina was varied up to 30% by weight. The composites were prepared using a two-roll mill and then test specimens were prepared by injection molding. Thermal conductivity, hardness, impact strength and melt flow rate of the composites increased with decreased particle sizes and increased particle content with exceptions at certain concentrations due to non-uniform distributions of particles and agglomerates formed by the particles. As an example, the best integrated thermal conductivity was shown by a 75 micron-Al₂O₃/HDPE composite at 15% wt. alumina content, while the 212 micron-Al₂O₃/HDPE composite at 20 wt.% alumina content. For the same alumina content of 15% concentration by weight out of the three particle sizes. 75, 212 and 850 microns, the 75 micron-Al₂O₃/HDPE composite gave the highest thermal conductivity, which was nearly 50% higher than that of pure HDPE. Enhancement in impact strength and Hardness Rockwell were up to 300% and 400% as compared to the pure HDPE respectively. The Al₂O₃ with small particle size is generally more efficient for the enhancement of the impact strength.

Keywords: HDPE, Thermal Conductivity, Impact Strength, Melt Flow Index, Hardness, Filler, Alumina

1. Introduction

An ever-increasing research interest has been shown in recent years by experts from both the industry and academia toward polymer-matrix composites, particularly for applications in the areas of aerospace equipment design, automotive construction, electronic devices, medical and pharmaceutical products, civil construction materials, chemical industries, and other consumer applications. This is due to the superior properties exhibited by these polymer composite materials such as high strength to weight ratio, excellent electrical conductivity/insulation, and of course good compatibility options coupled with easy and inexpensive processing [1-10].

In polymer composites, the matrix phase consisting mostly of the polymer remains the primary phase, which is more ductile phase and it holds the reinforcement that is the secondary phase. These reinforcements (mostly inorganic materials) are usually stronger than the polymer matrix, and

the resulting synergic effect of incorporating them into the polymer matrix result in enhanced mechanical properties of the polymer composite. Appropriately engineered design of the polymer composite material is a key to the resulting improved properties of the new combined material, which must be better than the virgin polymer matrix [1, 2, 5-7, 10].

Composite materials can be classified in different ways. Broadly, polymer composites can be classified into two groups on the basis of reinforcing material. They are fiber-reinforced polymer (FRP) and particle-reinforced polymer (PRP) composites. Fiber-reinforced polymer materials consist of fibers, which have high strength and modulus. Fibers can be bonded to the matrix, the interface that is boundary between the fiber and the matrix, which has different physical and chemical identities. Particle-reinforced polymer composites used for reinforcing include ceramics, small mineral particles, metal powders such as aluminum and

amorphous materials including polymers and carbon black. Particles are used to increase the modulus and to decrease the ductility of the matrix. They are also used to reduce the cost of the composites.

In many applications there is need to improve the properties of polyethylene, being the most commonly used plastic substance, in particular with respect to heat deformation resistance, but improvement in chemical resistance, stress cracking, etc. may be important as well. In cable jacketing, however in many occasions especially for low-voltage application (<10kv) the mechanical and thermal-endurance become more important than electrical properties of the material. For example a furnace cable operated at 500v requires good mechanical properties and stability of mechanical properties at elevated temperature which after aging is the most important consideration. On the other hand, for oil-resistant and fire retardant cables, the requirements are quite different.

In addition to polyethylene, nowadays vinyl acetate modified polyethylenes are also used in the wire and cable industry. The incorporation of small amount of vinyl acetate into polyethylene results in a resin which extrudes much like polyethylene while providing the ability to accept filler loading e.g. alumina [11]. Actually a wide range of properties is possible depending on the vinyl acetate content. Density, crystallinity and toughness can be changed by vinyl acetate. Another important requirement for cable in some applications is flame resistance. Due to corrosiveness and toxicity of smoke and other emission products generated during the combustion of thermoplastics containing halogen-based fire retardants, extensive works have been carried out to use the alternative halogen free fire retardants.

In particular some metallic hydroxides are gaining increasing importance in the industry because of the desirable combination of low cost, low smoke and relatively high fire retardant efficiency. Polymers with excellent electrical insulation have been extensively used as packing materials in electrical devices due to their good process ability, light weight and low cost. Nevertheless, the thermal conductivity of polymers are generally very low and it has been widely recognized that the thermal conductivity of these polymers has to be enhanced to lower the energy loss and increase the stability of the devices. With the increasing demand for high density power and energy transmission of electronic devices, fabrications of insulating polymer materials with high thermal conductivity has become crucial [5, 6, 11-13].

High crystallization and orientation of polymer can greatly enhance the thermal conductivity along the orientation direction, but they usually have difficulties of processing. Blending of polymer with inorganic fillers is an effective and convenient way to enhance the polymer thermal conductivity while maintaining the electrical insulation, such as polymer composites with boron nitride, aluminum nitride silicon carbide composites. The thermal conductivity increases with filler content. Very high filler loading is often used to obtain high thermal conductivity. However, it seriously alters the polymer component and can form an agglomerate which

induces stress concentration and decrease of the material, so the practical application of thermal conductive polymer is limited. Filler size and shape are also important factors for the thermal conductivity and mechanical properties. Alumina Al_2O_3 was used to fill silicone rubber by Zhou et al. [14] and reported that nano-sized Al_2O_3 composite exhibited higher thermal conductivity and mechanical properties than the micro-sized one. The influence of hybrid fillers was also investigated [15]. However, reports on how to enhance the thermal conductivity and mechanical properties of the polymer composites simultaneously are still quite limited, the size and content in the composite determine the average inter particle distance which is closely related to the thermal conductivity and mechanical properties. At the same particle content, smaller particle size leads to lower inter particle distance and more chances for the formation of thermal conductive pathway. The particle size and content affect the inter particle distance and the stress state of the matrix polymer surrounding the voids when the average inter particle distance is in a suitable range extensive plastic deformation in the matrix can be easily induced. So the optimization of the particle size and content can be a convenient and feasible way to prepare composites with good synthetic properties [15-19].

In this paper, Al_2O_3 /HDPE composites with different alumina particle sizes (diameter of 75 μ m, 212 μ m and 850 μ m) were prepared. The influence of alumina content and particle size on thermal conductivity impact strength, Hardness and Melt flow index (MFI) are studied. The synthetic properties of the composites are optimized and the mechanism is also discussed.

2. Experimental

2.1. Materials

Injection grade high density polyethene HDPE (M 60075) was supplied by Eleme Petrochemicals Limited Port Harcourt having density of 0.960g/cm³ and melt flow index MFI of 8.2g/10mm (1900c, 2.16kg). Alumina (Aluminum III Oxide) was supplied by Vickers Laboratory LTD Burley in Whaefedale West Yorkshire England. The Alumina was passed through various sieves. The three grades of particle sizes selected as fillers were:

1. Particles that passed through sieve with cell diameter of 75 microns
2. Particle that passed through sieve with cell diameter of 212 microns
3. Particles that passed through sieve with cell diameter of 850 microns.

All of the alumina particles were of crystalline nature and the thermal conductivity was about 30 W/(m.k)

2.2. Sample Preparation

144g of each grade of alumina were measured using electronic balance and five different concentrations were calculated viz: 10%, 15%, 20%, 25% and 30% respectively by weight. Each concentration of alumina particles for the three

grades, 75microns, 212microns and 850microns were added to the 144 g of pre-weighed HDPE without surface pretreatment.

The HDPE-Alumina composites were prepared using a two-roll mill X(S) K-140, Lord Mashal Group Company, Onitsha for 30mins. The temperature of the front roll and back roll were 120 and 110°C respectively. The extrudates were granulated and the granules for each particle size of Alumina with the respective concentrations by weight was fed into injection molding machine of 120kg/cm³ capacity at 190°C together with five different colorants viz: Red, Green, Gold, Blue and Orange to distinguish between different particle sizes of alumina and various concentrations by weight. The mold was designed to produce a mirror cover which was used as the prepared test Specimen. Weight of each shot was about 25g. Barrel temperature set points of rear zone, middle zone and nozzle zone were 1000c, 150 and 1900 respectively. The sample of HDPE composites prepared by injection molding was kept at room temperature for 48hours prior to testing to promote relaxation of stress.

2.3. Characterization

2.3.1. Thermal Conductivity

The sample for thermal conductivity measurement was cut into rectangular bars. The test was conducted according to ASTM E225-09, standard test method for thermal conductivity. The mass m, length L, and the thickness X of specimens of various concentrations of HDPE – Alumina concentrations were determined. The temperature change was determined by taking room temperature as the initial temperature and final temperature gotten after period in thematically controlled oven. The specific heat capacity of each specimen was gotten from the relation.

$$H = MC\Delta\theta \quad (1)$$

Where H = IVt, IV is the power rating of the oven, t is the time material stayed in the oven, M = Mass and C is the specific heat capacity.

The thermal conductivity was finally obtained from the relation

$$K = HX/A\theta \quad (2)$$

where K is the thermal conductivity; A is the area of the material.

2.3.2. Hardness Survey Test – (ASTM D 785)

Hardness survey test was carried out through procedure for hardness measurement using hardness Testing Machine with Test Method – 160 6508, 160 716, procedure 200 M014 Rock well scale B. The Allen Screw in pressure shaft below the front panel was released and the indenter was put into the pressure shaft so that flat part of indenter cylinder was in front of Allen Screw. Allen screw was slightly tightened to eliminate gap in forcing system after which the machine main

switch in front of the panel was switched on. A load of 60kg was selected as the loading force on the right side of the equipment. Anvil under the indenter was lowered down enough and the test specimen was put on it making the testing place directly against indenter. The anvil and the sample specimen were lifted by the use of lifting screw until the green light in front panel was on which marked the starting of the test. The sample specimen was released when red light signifying the end of the test was shown. The procedure was repeated on another spot of the testing plane and the average hardness value was taken.

2.3.3. The Impact Strength Test

The impact strength of the prepared specimens was determined using charpy impact test. Pendulum-testing machine was used for the test with test method - 150 148, procedure 200 M 014 charpy impact. The samples were prepared by marking the position for impact test on the test specimen, before it was properly placed on the anvil. The specimens were broken by a single overload event due to the impact of the pendulum. A stop pointer was used to record how far the pendulum swung back up after fracturing the specimens in the scale. The impact strength was determined by measuring the energy absorbed in the fracture of the specimen. The energy absorbed was determined by noting the height at which the pendulum was released and the height to which the pendulum swung after it has struck the specimen. The height of the pendulum times the weight of the pendulum produced the potential energy and the difference in potential energy of the pendulum at the start and the end of the test is equal to the absorbed energy.

3. Results and Discussion

It was visually observed upon experimental studies that the addition of alumina in HDPE made the melt more viscous. It also made processing difficult requiring high temperature and more injection pressure. The density of the composite increased with increasing alumina concentration.

Thermal Conductivity

The variation of thermal conductivity against concentration of alumina particles in the Al₂O₃/HDPE polymer composite was investigated in the range 0 till 30% wt/wt for particles of three different sizes, 75 μm, 212 μm, and 850 μm (Table 1). It was generally observed that the thermal conductivity increased gradually with increment in the filler content up to 15% concentration and experienced decrease at 20% and 25% concentrations by weight before gradually increasing up to the concentration of 30% composites. The fluctuations in the values of thermal conductivity as the concentrations by weight increases is due to non-uniform distribution of the particle as the composites pass through the injection machine [20.21].

Table 1. Thermal conductivity for Al₂O₃/HDPE composites at different concentrations and Al particle sizes.

Particle size	75 μm			212 μm			850 μm		
	Alumina concentration in composite, wt. %	Mass, (Kg)	Thickness, (m)	Thermal conductivity (kw/m°C)	Mass, (Kg)	Thickness, (m)	Thermal conductivity (kw/m°C)	Mass, (Kg)	Thickness, (m)
10	0.0023	0.00136	0.00292	0.0023	0.00135	0.00290	0.0025	0.00135	0.00315
15	0.0025	0.00130	0.00303	0.0021	0.00128	0.00251	0.0025	0.00122	0.00285
20	0.0023	0.00137	0.00294	0.0025	0.00139	0.00324	0.0021	0.00135	0.00269
25	0.0020	0.00132	0.00246	0.0022	0.00126	0.00259	0.0023	0.00129	0.00277
30	0.0023	0.00132	0.00283	0.0024	0.00134	0.00300	0.0024	0.00137	0.00412
HDPE	0.0020	0.00130	0.00200	0.0020	0.00130	0.00200	0.0020	0.00130	0.00200

Table 2. Impact strength test for Al₂O₃/HDPE composites at different concentrations and Al particle sizes.

Particle size	75 μm		212 μm		850 μm	
	Alumina concentration in composite, wt. %	Energy Absorbed (J)	Mol for Unnoticed Beam	Energy Absorbed (J)	Mol for Unnoticed Beam	Energy Absorbed (J)
10	4.8	0.00109	4.0	0.00091	2.2	0.00050
15	2.4	0.00055	2.6	0.00059	4.7	0.00107
20	4.6	0.00105	3.0	0.00068	4.6	0.00105
25	2.3	0.00052	2.0	0.00045	2.8	0.00064
30	2.3	0.00052	4.2	0.00095	2.6	0.00062
HDPE	2.0	0.00040	2.0	0.00040	2.0	0.00040

It was observed that at 10% concentration by weight of Alumina particle, thermal conductivity increased but as the concentration by weight increased to 15% and 20%, there was a gradual decrease in the thermal conductivity. However, the increase in filler content up to 25% and 30% concentration by weight led to rapid increase in the thermal conductivity of the HDPE/Alumina composite.

Table 3. Hardness survey test for Al₂O₃/HDPE composites at different concentrations and Al particle sizes.

Particle size	75 μm			212 μm			850 μm		
	Alumina concentration in composite, wt. %	HR-1	HR-2	MEAN-HR	HR-1	HR-2	MEAN-HR	HR-1	HR-2
10	461	475	468	655	526	591	476	472	474
15	512	467	490	861	701	781	624	580	602
20	610	535	573	582	527	555	554	619	587
25	816	824	820	668	767	672	619	510	565
30	515	526	521	710	995	853	621	670	691
HDPE	234	202	218	234	202	218	234	202	218

+ The example for this table.

Melt Flow Index

The melt flow index result for different HDPE/Alumina Particle composites is shown on Table 4. The melt flow index of the pure sample of high density polythene (HDPE) at the temperature of 1900c is 10.6. On the subsequent addition of 10%, 15%, 20% and 25% concentration by weight of the alumina particle, it was discovered that the melt flow index of the HDPE/Alumina composites increased. The increment is in order of the increase in the percentage composition of alumina particle.

Mechanical Properties of the Composite

Impact Strength

The variations of impact strength of composite with alumina volume content are presented in Table 2.. It can be deduced from the Table that the impact strength increases with increase in the alumina content. The toughening efficiencies of the alumina particles are dramatically different. There are several mechanisms of toughening of polymer for the inorganic particles toughening polymer, at least three factors are necessary. Inherent ductility of the matrix deboning and suitable inter-particle distance. The stress

concentration first leads to deboning of the filler particles and void formation. The particle content affects the inter-particle distance and the stress state of the matrix polymer surrounding the voids [22-24]. At low alumina content, the inter-particle distance was long and the inter-particle matrix ligament lies in plane strain state which is hard to yield, as a result, the impact strength was not improved. When the alumina content was increased up to 10% weight concentrations for 75μm, 212μm and 850μm alumina particle sizes, the inter-particle distance reached a suitable range, the inter-particle matrix ligament lied in press state, which can be plastic yield easily and the impact strength was improved. But when the alumina content was too high, the inter-particle distance became too small and led to large size agglomerates which provided convenient triggers for brittle behavior. When the alumina content was 10% by weight concentration for 75μm, 212μm and 850μm, the surfaces were generally even and smooth and the plastic yield increased. The increase of particle content decreased the average inter-particle distance of the matrix and resulted in the apparently plastic yield which led to high impact strength of the composites.

From the graph of the impact strength against the particle sizes, it can be deduced that HDPE/Alumina composites of 75 μ m alumina particle size gave higher impact strength and effective toughening for the same weight concentration than 212 μ m alumina particle size and 212 μ m alumina particle size gave higher impact strength and effective toughening than 850 μ m alumina particle size but as the concentration of such small grade particle size increases, the inter-particle distance becomes too small which leads to the large particle agglomerates, from the graphs of impact strength against different concentrations by weight for 75 μ m, 212 μ m and 850 μ m alumina particle. It was deduced that the impact toughness of the various concentrations by weight of HDPE/Alumina composites for 75 μ m, 212 μ m and 850 μ m were determined by measuring the energy absorbed in the fracture of the specimen. The energy absorbed is highest for alumina particle size of 75 μ m and increases according to the concentration by weight of the particle size. The energy absorbed by 212 μ m particle size was higher than 850 μ m particle size and they all increased with increase in the concentration by weight. As the increase in the energy absorbed in proportional to the impact toughness, it follows that the impact toughness increases with decrease particle size and increased particle content. The energy absorbed by 10% 75 μ m is about 2.5 times higher than the energy absorbed by HDPE, while 10% 212 μ m absorbed energy exactly twice the energy absorbed by HDPE and 10% 850 μ m absorbed energy 0.5 times greater than absorbed by HDPE.

The Effect of Alumina Particle Size and Content on the Hardness of HDPE-Alumina Composite

Hardness is the resistance of a material to deformations, indentation or scratching. For the purpose of this study, Hardness Rockwell Test was performed to measure the relative hardness of the material. The method was based on the rate of penetration of a specified indenter force into the material under specified condition and the sample placed on a flat surface. Each sample was subjected to Rockwell Hardness Test at different positions on the sample base and the average value for each sample was recorded in accordance with ASTM D 785. The mean values of Rockwell hardness against different alumina particle sizes of varying concentrations by weight are shown in Table 3. From the result obtained, Alumina-HDPE composite showed a high hardness value when compared to the unfilled system. The hardness value increases with the increase in alumina content. For 75 μ m alumina particle size of concentrations by weight of 10%, 15%, 20%, 25% and 30%, the mean Rockwell hardness value comply with the following order 468 < 490 < 573 < 820 > 521 respectively. The sudden decrease in the mean Rockwell hardness at 30% concentration by weight is attributed to the average inter-particle distance being too small to cause agglomerate of the particle at a particular point leading to non-uniform distribution of the particles in the matrix [5, 25-28]. Similar trend of increment was observed for particle sizes of 212 μ m and 850 μ m of various concentrations by weight. Apart from the fluctuations for the obvious reason afore-mentioned the size of the particle

equally had a great influence on the Mean Rockwell Value of the Alumina/HDPE composites. The Rockwell hardness value is highest for 75 μ m particle size followed by 212 μ m particle size and then 850 μ m particle size considering 20% concentration by weight of 75 μ m, 212 μ m and 850 μ m with Rockwell mean values of 820, 672 and 565 respectively proving that the smaller the particle size, the higher the hardness value of the composite.

4. Conclusion

In this study, the thermal conductivity and mechanical properties (impact strength and hardness of three types of Al₂O₃/HDPE composites with alumina filler sizes of 75 microns, 212 microns and 850 microns were investigated. The alumina particle with smaller particle size can form thermal conductive alumina pathway more easily and has higher toughening efficiency for 75 microns and 212 microns. However, the smaller the particle size, the greater chances of the formation of aggregation and that destroys the toughness of the composites. A 75 microns/HDPE at alumina content of 15% owns the best integrated thermal conductivity. A 212 microns/HDPE at alumina content of 20% owns the best integrated thermal conductivity while 850 microns/HDPE at alumina content of 30% showed the highest thermal conductivity. For the same alumina content of 15% concentration by weight for 75 microns, 212 microns and 850 microns, 75 microns particle size resulted to the highest thermal conductivity. Its thermal conductivity is nearly 1.5 times of the pure HDPE.

The impact strength for 212 microns was highest at 30% concentration by weight and highest at the 15% concentration by weight for 850 microns alumina particles. At 25% concentration by weight 75 microns alumina particles, Hardness Rockwell was highest. The impact strength and Hardness Rockwell are about 3 and 4 times of the pure HDPE respectively. This study would provide practical and theoretical supports for thermal conductive polymer composites with good synthetic properties.

Table 4. Melt flow index (MFI) of Al₂O₃/HDPE composites at different concentrations.

Alumina concentration in composite, wt. %	MFI
10	13.2
15	15.4
20	17.2
25	19.0
HDPE	10.6

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