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Studies on effects of metakaolin as a filler in some commercial thermoplastics polymers

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ABSTRACT

Particulate filled polymer composites are becoming attractive because of their wide applications and low cost. In this study the effects of metakaolin (Surface treated & non-treated) with varying concentration on the mechanical properties of some commercial thermoplastic polymers was investigated. Composites of low density polyethylene, high density polyethylene and polypropylene with varying concentrations (viz. 5 to 30 weight %) of metakaolin were prepared by screw extrusion. Mechanical properties such as tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength were found to increase with metakaolin concentration. Morphological studies revealed that there is a good dispersion of metakaolin in the polymer matrix.

Key words: Metakaolin, thermoplastics, composite, mechanical properties

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INTRODUCTION

Pioneers of additives realized that their chemistry offers multiple advantages for use in plastics. Various findings regarding additives and their applications, have led to tremendous growth as reflected by their usage in plastics processing and end-product applications. The performance of filled polymers is generally determined on the basis of the interface attraction of filler and polymers. Incorporating inorganic mineral fillers into plastic resin improves various physical properties of the materials such as mechanical strength, modulus etc. In general the mechanical properties of particulate filled polymer composites depend strongly on size, shape and distribution of filler particles in the polymer matrix and extent of interfacial adhesion between filler and matrix. Most commonly used fillers in making composites include metal oxide, metal powders, carbon black, silica, spherical or fibrous glass, talc, calcite, 3,4 kaolin, mica etc. Particulate fillers may have different shapes such as spherical, spheroidal, or cubic. Fillers are directly used in untreated or chemically treated forms prior to compounding with various thermoplastics. There are many beneficial reports on chemically treated fillers that show a better adhesion with polymer matrix than that of untreated ones.

Almost 85% of the polymers produced worldwide are thermoplastics [1]. Melting temperatures can be as high as 365 ^{o}C for polyetherketone (PEK), as low as 110 ^{o}C for low-density polyethylene (LDPE), and even lower for

ethylene–vinyl acetate (EVA) copolymers [1]. Over 70% of the total production of thermoplastics is accounted for by the large volume, low-cost commodity resins: polyethylenes (PE) of different densities, isotactic polypropylene (PP), polystyrene (PS), and PVC. Next in performance and in cost are acrylics, acrylonitrile–butadiene–styrene (ABS) terpolymers, and high impact polystyrene (HIPS). Engineering plastics such as acetals, polyamides, polycarbonate, polyesters, polyphenylene oxide, and blends thereof are increasingly used in high-performance applications. Specialty polymers such as liquid-crystal polymers, polysulfones, polyimides, polyphenylene sulfide, polyetherketones, and fluoropolymers are well established in advanced technology areas because of their high Tg or Tm (290–350 ^{o}C).

Global demand for fillers/reinforcing fillers including calcium carbonate, aluminium trihydrate, talc, kaolin, mica, wollastonite, glass fiber, aramid fiber, carbon fiber, and carbon black for the plastics industry has been estimated to be about 15 million tons [2]. Primary end-use markets are building/construction and transportation, followed by appliances and consumer products; furniture, industrial machinery, electrical/electronics, and packaging comprise smaller market segments. Flexural modulus and heat resistance are the two critical properties of plastics that are enhanced by the inclusion of performance minerals. Automotive exterior parts, construction materials, outdoor furniture, and appliance components are examples of applications benefiting from enhanced flexural modulus. Automotive interior and underhood parts, electrical connectors, and microwaveable containers are examples of applications requiring high-temperature resistance. Environmental acceptance and improved sustainability of automotive parts are attributes of composites containing natural fibres. Life cycle assessment studies [3] tend to support the position that natural fibre composites are environmentally superior to glass fibre composites. Recent Statistics estimate the U.S. demand for fillers and extender minerals to a total of 3.2 million tons per annum [4]. Annual growth rates are estimated to be 2–3% with much higher rates for fire retardant fillers such as aluminium hydroxide (5.5–7%). Data (not including glass products and natural fibres but including TiO_2 and organoclays) indicate the highest demand for ground calcium carbonate followed by TiO_2 and aluminum trihydrate. Talc, kaolin, mica, wollastonite, silica, barites, and organoclays have a much smaller share of the market.

Increased polymer consumption over the past 20 years has not only stimulated machinery sales but also led to a parallel growth in the usage of a large variety of liquid and solid modifiers including fillers and reinforcements [5]. Significant advances have been made to accommodate such additives by improving the efficiency of polymer mixing/compounding equipment. Thermoplastic resin compounders combine the polymer(s) with the modifiers in high-intensity batch mixers and continuous extruders (mostly twin-screw extruders), and the material is then pumped into a pelletizer to produce the feed for subsequent shaping operations. Thermosetting resin suppliers compound heat-sensitive resins with fillers, additives, and/or pigments in a variety of mixers to produce molding compounds in such forms as powder, granules, and pastes to be fed into the molding equipment.

There are a significant number of technological advances that will undoubtedly contribute to the additional growth in the usage of certain functional fillers: for example, for wood-filled plastics, introduction of specially configured counter-rotating twin-screw extruders with vent zones to remove moisture [6]; for mica and talc, development of new grinding technologies to retain the lamellarity and aspect ratio of the plate-like fillers [7] and for other minerals to produce ultrafine particles by special grinding methods [8]; for deagglomeration, dispersion, and exfoliation of agglomerated nanoclays particularly in high-temperature thermoplastic matrices, clay modification with additives such as ionic liquids [9] having higher thermal stability than the existing alkylammonium modifiers; in addition, melt compounding in extruders with improved screw configuration, and optional ultrasonic assistance or the use of supercritical fluids; for carbon nanotube composites, appropriate interfacial modification to improve dispersion and adhesion and minimize deagglomeration; also, equipment/process modification to ensure the desired orientation and maintain the high aspect ratio.

Some new exciting application areas for composites containing cationic or anionic nanoclays, nano oxides, carbon nanotubes, ultrafine TiO2, talc, and synthetic hydroxyapatite are

1. structural materials with improved mechanical, thermal, and barrier properties, electrical conductivity, and flame retardancy;
2. high-performance materials with improved UV absorption and scratch resistance;
3. barrier packaging for reduced oxygen degradation;
4. multifunctional fillers that could release in a controlled manner corrosion sensing additives, corrosion inhibitors, insecticides, active pharmaceutical ingredients, and so on;

5. bioactive materials for tissue engineering applications.

MATERIALS AND METHODS

The materials used in this investigation are as following:
Low density polyethylene, High density polyethylene and Polypropylene are obtained from Reliance Industries Limited of moulding grade. Different grades of Metakaolin are obtained from Shree Ram Mineral, Bhuj, Gujarat and all other chemicals used were of LR grade.

Four different grades of m-kaolin used and details are given as follows.
- 61: Grade Name: Meta Kaolin (White), C/S R.F. No. 280513/61
- 57: Grade Name: Kaofil – Y, C/S R.F. No. 280513/57
- 55: Grade Name: Calcined Kaolin – 0.15, C/S R.F. No. 280513/55
- 54: Grade Name: Kaofil – 82, C/S R.F. No. 280513/54

2.2 Surface Modification of Metakaolin
Prior to surface treatment m-kaolin is dried in oven at 50 ^{0}C for 1 hour to removed absorbed moisture. By treatment of metakaolin with NaOH solution with continuous stirring for 2 hrs and was left for one day. Afterwards excess of NaOH solution was removed. Then it was neutralized and was given acidic treatment with H_2SO_4 solution (50%) with continuous stirring for 2 hrs and was then kept for a day. Afterwards excess of acid was removed. Now we got a suspension which couldn't be separated by just gravity sedimentation so it was then subjected to ultracentrifugation at 7000 rpm. The metakaolin got was dried in oven at 100 ^{o}C for a day. Hence the metakaolin had been modified and called as **treated metakaolin,**

2.3 Blending of filler with different thermoplastic polymers:
The polymers used for the blending were moulding grade of polypropylene, high density polyethylene and low density polyethylene. Four different grades of m-kaolin are used as filler. Both treated and untreated fillers dried in oven at 90°C for 24 h before blending to remove moisture. The effect of filler loading after and before treatment was studied making use of the different blend compositions. The blending was performed using a single screw extruder. The temperature of each zone was set at T1 = 60 ^{0}C, T2 = 150 ^{0}C, T3 = 200 ^{0}C (for polypropylene), T1 = 60 ^{0}C, T2 = 150 ^{0}C, T3 = 190 ^{0}C (for HDPE), T1 = 60 ^{0}C, T2 = 140 ^{0}C, T3 = 170 ^{0}C (for LDPE). The extrudate was cooled in water, dried at 80-90 ^{0}C in oven and was then ground.

2.4 Batch formulation:
Polypropylene (PP):

Sr. No.	Batch Size in gm	Polyprolylene %	Filler %	Polypropylene gm.	Filler gm.
1.	300	98	2	294	6
2.	300	94	4	288	12
3.	300	90	10	270	30
4.	300	85	15	255	45
5.	300	80	20	240	60
6.	300	75	25	225	75

Batch formulation for High Density Polyethylne (HDPE) and Low Density Polyethylne (LDPE) were prepared same as batch prepared for Polypropylene (PP).

2.5 Test Specimen Preparation:
To prepare standard test specimen for tensile, impact and flexural strength measurement, automatic injection moulding machine is used. Dumbbell shaped tensile specimens, bar shaped flexural strength specimens and impact strength specimens of the different blends were prepared. The temperature for each zone was set at T1 = 60^0 C, T2 = 90 ^{0}C, T3 = 150 ^{0}C, and T4 = 215 ^{0}C (for polypropylene), T1 = 60 ^{0}C, T2 = 100 ^{0}C, T3 = 150 ^{0}C, and T4 = 200 ^{0}C (for HDPE), T1 = 60 ^{0}C, T2 = 80 ^{0}C, T3 = 140 ^{0}C, and T4 = 180 ^{0}C (for LDPE). The cooling time must be same for all the test specimens. After ejection of the sample, the flash is removed manually by the use of cutter.

2.6 Mechanical and Physical Test:
Conditioning (ASTM D 618)
A true material comparison is possible only when property values are determined by identical test methods under identical conditions [10]. Generally speaking, physical and electrical properties of plastics and electrical insulating materials are affected by temperature and humidity. Plastic materials tested above room temperature will yield relatively higher impact strength and lower tensile strength and modulus. High humidity tends to alter the electrical property test results. Obviously, in order to make reliable comparisons of different materials and test results obtained by different laboratories, it is necessary to establish standard conditions of temperature and humidity.

Conditioning is defined as the process of subjecting a material to a stipulated influence or combination of influences for a stipulated period of time [11]. Three basic reasons for conditioning specimens are:

- To bring the material into equilibrium with normal or average room conditions
- To obtain reproducible results regardless of previous history or exposure
- To subject the material to abnormal conditions of temperature and humidity in order to predict its service behaviour

Standard Laboratory Temperature: Standard laboratory temperature is defined as 23 °C (73.4 °F) with a standard tolerance of +2 °C (+3.6 °F).

Standard Laboratory Atmosphere: Standard laboratory atmosphere is defined as an atmosphere having a temperature of 23 °C (73.4 °F) and a relative humidity of 50 percent with a standard tolerance of +2 °C (+3.6 °F) and +5 percent, respectively.

2.7 Measurements:
Mechanical Testing
The mechanical properties, among all the properties of plastic materials, are often the most important properties because virtually all service conditions and the majority of end-use applications involve some degree of mechanical loading. Nevertheless, these properties are the least understood by most design engineers. The material selection for a variety of applications is quite often based on mechanical properties such as tensile strength, modulus, elongation, and impact strength. In practical applications, plastics are seldom, if ever; subjected to a single, steady deformation without the presence of other adverse factors such as environment and temperature. Since the published values of the mechanical properties of plastics are generated from tests conducted in a laboratory under standard test conditions, the danger of selecting and specifying a material from these values is obvious. A thorough understanding of mechanical properties, tests employed to determine such properties, and the effect of adverse conditions on mechanical properties over a long period is extremely important.

Tensile Tests (ASTM D 638) [12, 13]
Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress–strain diagram. Different types of plastic materials are often compared on the basis of tensile strength, elongation, and tensile modulus data.

Flexural Properties (ASTM D 790) [14, 15]
The stress–strain behavior of polymers in flexure is of interest to a designer as well as a polymer manufacturer. Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. The stresses induced by the flexural load are a combination of compressive and tensile stresses.

Izod Impact Test (ASTM D-256, ASTM D 4812) [16]
The objective of the Izod impact test is to measure the relative susceptibility of a standard test specimen to the pendulum-type impact load. The results are expressed in terms of kinetic energy consumed by the pendulum in order to break the specimen. The energy required to break a standard specimen is actually the sum of energies needed to deform it, to initiate its fracture, and to propagate the fracture across it, and the energy expended in tossing the broken ends of the specimen. This is called the “toss factor.” The energy lost through the friction and vibration of the apparatus is minimal for all practical purposes and usually neglected.

RESULTS AND DISCUSSION

Mechanical properties polymer composites were measure for all different grades of fillers discussed in previous chapter. Results are given in Table No. 1 to Table No. 6 and following conclusions are made:

Table: 1 Tensile Properties of Polypropylene with various percentage of filler

Polymer + Filler code	% of filler loading	Tensile Strength Kgf/(cm)^2		Elongation (%)	
		Treated	Non-Treated	Treated	Non-Treated
PP	0	230.72		38	
PP + 61	2	240.72	233	37	38
	4	242.89	233.78	32	36
	10	254.48	245.73	30	35
	12	291.46	243.52	29	32
	15	281.03	245.73	28	30
	20	282.16	248.39	26	28
	25	253.16	246.51	22	25
PP + 54	2	240.21	232.5	36	37
	4	242.38	233.28	33	35
	10	253.97	244.23	30	33
	12	290.95	243.02	27	30
	15	280.52	245.53	25	29
	20	281.65	244.89	24	23
	25	252.65	241.01	23	23
PP + 55	2	237.11	232.1	36	36
	4	239.28	232.88	34	35
	10	250.87	243.83	32	34
	12	287.85	242.62	31	34
	15	277.42	245.13	28	32
	20	278.55	244.49	26	30
	25	249.55	240.61	25	26
PP + 57	2	233.01	231.40	36	37
	4	235.18	232.18	34	35
	10	246.77	243.13	31	31
	12	283.75	241.92	29	30
	15	273.32	244.43	26	29
	20	274.45	243.79	24	26
	25	245.45	239.91	22	22

Thermoplastics polymer such as polypropylene, high density polyethylene (HDPE), and Low density polyethylene (LDPE) were used as a polymer matrix in making particulate composites.

Fillers were used without stearic acid coating on their particle. All thermoplastics polymer composites batch contain filler content is 2%, 4%, 10%, 12%, 15%, 20% and 25%. Dispersion of fillers achieved in different batches by single screw extruder in presence of 1% of silicon oil as a processing aid.

Polymer blend are prepared by surface treated and non surface treated fillers containing same percentage loading. All four thermoplastics material, dispersion of fillers obtains upto 25% and by increasing the percentage of fillers produces voids in the prepared sample. Higher percentage of fillers also leads to phase separation. This problem can be solved by creating stearic acid coating on particle. Stearic acid reduces the molecular interaction of the polymer molecules by reducing Vander walls forces.

Mechanical properties such as tensile strength, % elongation, flexural strength and impact strength were measured and calculated. Observation of results shows good dispersion of fillers into polymer matrix and also gives increase in mechanical properties. Results of tensile strength for surface treated fillers show that: In polypropylene material gives maximum tensile strength about 234 KgF/cm^2 for filler number 61 & 54 while filler number 55 and 57 about 221 KgF/cm^2 and 214 KgF/cm^2. In HDPE material filler number 61 & 57 gives about 195 KgF/cm^2 while filler number 54 & 55 gives about 162 KgF/cm^2. In LDPE material filler number 61 gives 95 KgF/cm^2 and other fillers give about 110 KgF/cm^2. The non treated fillers give upto reduction in results up to 10 – 15%. Surface treatment of fillers makes more dispersion but at the same time after acid and base treatment maintaining of pH is very important

otherwise it leads to corrosion to the processing machinery. Flexural strength results also gives good effects of filler dispersion. Filler number 61 & 54 give good results in polypropylene, HDPE and polycarbonate material while in LDPE material all fillers have similar type of values. Details of values are given in results table. Impact strength is not highly affected as the percentage of filler increase. Generally the impact strength values are reduces but in all four fillers it is not too reduces.

Table: 2 Tensile Properties of High Density Polyethylene (HDPE) with various percentage of filler

Filler code	% of filler loading	Tensile Strength Kgf/(cm)^2		Elongation (%)	
		Treated	Non-Treated	Treated	Non-Treated
HDPE	0	202.64			
HDPE + 61	2	212.64	204.92	84	72
	4	214.81	205.70	83	71
	10	226.40	217.65	82	70
	12	263.38	215.44	72	64
	15	252.95	217.65	66	60
	20	254.08	220.31	64	56
	25	225.08	218.43	60	54
HDPE + 54	2	212.13	204.42	96	73
	4	214.30	205.2	95	72
	10	225.89	216.15	94	72
	12	262.87	214.94	88	69
	15	252.44	217.45	70	3866
	20	253.57	216.81	62	64
	25	224.57	212.93	95	60
HDPE + 55	2	209.03	204.02	93	96
	4	211.2	204.8	90	92
	10	222.79	215.75	90	91
	12	259.77	214.54	85	85
	15	249.34	217.05	80	83
	20	250.47	216.41	74	78
	25	221.47	212.53	67	72
HDPE + 57	2	204.93	203.32	97	90
	4	207.1	204.1	92	84
	10	218.69	215.05	88	82
	12	255.67	213.84	85	80
	15	245.24	216.35	81	78
	20	246.37	215.71	77	73
	25	217.37	211.83	74	69

Table: 3 Tensile Properties of Low Density Polyethylene (LDPE) with various percentage of filler

Filler code	% of filler loading	Tensile strength Kgf/(cm)^2		Elongation (%)	
		Treated	Non-Treated	Treated	Non-Treated
LDPE	0	70.46			
LDPE + 61	2	74.39	71.80	177	149
	4	75.56	72.43	172	138
	8	76.13	73.23	160	126
	12	76.96	74.05	131	112
	15	77.24	75.70	109	98
	20	83.45	80.64	105	88
	25	94.16	88.19	98	78
LDPE + 54	2	72.20	71.51	182	160
	4	73.40	72.31	172	146
	8	74.50	73.20	156	134
	12	75.51	74.32	132	126
	15	79.83	76.20	112	118
	20	89.93	83.39	109	108
	25	103.12	95.36	101	100
LDPE + 55	2	73.50	71.24	183	177
	4	74.76	72.87	176	166
	8	74.83	73.23	163	155
	12	76.16	74.77	144	148

	15	82.29	78.25	142	136
	20	94.45	90.52	134	132
	25	107.52	103.10	122	120
LDPE + 57	2	72.24	71.50	184	180
	4	73.56	72.62	178	175
	8	74.19	73.41	165	163
	12	78.88	74.16	156	154
	15	86.66	83.29	144	142
	20	98.25	93.11	136	133
	25	110.11	101.23	123	122

Table: 4 Flexural & Impact Properties of polypropylene (PP) with various percentage of filler

Filler code	% of filler loading	Flexural Strength Kgf/(cm)^2		Impact Strength (Joule/cm)	
		Treated	Non-Treated	Treated	Non-Treated
PP	0	568.65		0.5	
PP + 61	2	570.15	569.23	0.51	0.51
	4	693.97	721.43	0.55	0.54
	10	756.71	757.78	0.63	0.62
	12	771.32	772.15	0.69	0.68
	15	776.27	782.65	0.77	0.75
	20	755.89	783.79	0.83	0.81
	25	750.76	785.92	0.88	0.86
PP + 54	2	569.78	569.54	0.52	0.51
	4	629.31	578	0.56	0.54
	10	727.4	595.29	0.64	0.62
	12	740.09	599.85	0.69	0.68
	15	797.61	607.61	0.76	0.74
	20	789.22	605.59	0.84	0.82
	25	781.35	601.58	0.89	0.87
PP + 55	2	569.16	569.32	0.51	0.51
	4	618.19	614.41	0.55	0.54
	10	638.52	717.73	0.63	0.62
	12	689.36	692.54	0.69	0.68
	15	724.55	643.7	0.75	0.74
	20	715.19	694.41	0.83	0.82
	25	703.33	663.37	0.88	0.87
PP + 57	2	568.89	568.92	0.51	0.51
	4	627.21	660.22	0.56	0.54
	10	654.99	662.86	0.64	0.62
	12	743.18	734.18	0.70	0.69
	15	765.02	755.28	0.76	0.74
	20	726.89	739.66	0.83	0.82
	25	712.93	718.62	0.89	0.88

Table: 5 Flexural & Impact Properties of low density polyethylene (LDPE) with various percentage of filler

Filler code	% of filler loading	Flexural Strength Kgf/(cm)^2		Impact Strength (Joule/cm)	
		Treated	Non-Treated	Treated	Non-Treated
LDPE	0	91.34		0.8	0.8
LDPE + 61	2	93.65	93.57	0.84	0.83
	4	94.89	96.41	0.92	0.91
	10	97.88	100.82	0.98	0.96
	15	100.76	104.47	1.06	1.04
	20	103.07	108.4	1.12	1.1
	25	107.19	113.26	1.17	1.15
LDPE + 54	2	93.25	93.11	0.83	0.78
	4	94.1	94.91	0.91	0.86
	10	96.98	99.68	0.97	0.91
	15	102.66	103.05	1.05	0.99
	20	102.59	107.47	1.11	1.05
	25	106.69	111.88	1.16	1.1
LDPE + 55	2	92.86	92.78	0.815	0.76
	4	94.4	95.89	0.895	0.84
	10	97.23	100.68	0.955	0.89
	15	100.09	104.12	1.035	0.97

	20	103.55	103.39	1.095	1.03
	25	107.07	113.46	1.145	1.08
LDPE + 57	2	92.68	92.57	0.84	0.82
	4	94.33	96.11	0.91	0.9
	10	97.24	100.77	0.98	0.97
	15	100.12	104.13	1.07	1.05
	20	103.14	108.39	1.13	1.1
	25	107.09	112.57	1.17	1.15

Table: 6 Flexural & Impact Properties of high density polyethylene (HDPE) with various percentage of filler

Filler No.	% Filler loading	Flexural Strength KgF/cm^2		Impact Strength (Joule/cm)	
		Treated	Non-Treated	Treated	Non-Treated
HDPE	0	362.72		0.6	
HDPE + 61	2	368.56	366.56	0.66	0.65
	4	492.38	460.06	0.73	0.72
	10	555.12	496.41	0.82	0.81
	15	569.73	510.78	1.05	1.02
	20	574.68	521.28	1.22	1.15
	25	554.3	522.42	1.4	1.33
HDPE + 54	2	366.15	365.23	0.65	0.64
	4	425.68	373.69	0.72	0.70
	10	523.77	390.98	0.82	0.80
	15	523.77	395.54	1.06	1.05
	20	581.29	403.3	1.23	1.21
	25	572.9	401.28	1.39	1.38
HDPE + 55	2	365.54	364.32	0.64	0.63
	4	414.57	409.41	0.71	0.70
	10	434.9	512.73	0.81	0.80
	15	485.74	487.54	1.05	1.06
	20	520.93	438.7	1.21	1.22
	25	511.57	489.41	1.38	1.39
HDPE + 57	2	364.54	363.56	0.65	0.64
	4	422.86	454.86	0.72	0.70
	10	450.64	366.2	0.82	0.80
	15	538.83	434.88	1.06	1.05
	20	560.67	384.66	1.23	1.21
	25	522.54	347.94	1.39	1.38

CONCLUSION

Stearic acid coating makes filler more dispersible and also it provides lubrication. Reductions in particle size also increase the filler loading. Application of twin screw extruder increases the filler content values.

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