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Journal of Engineering and Applied Science (2022) 69:112
https://doi.org/10.1186/s44147-022-00166-5

Journal of Engineering and Applied Science

RESEARCH

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Analysis of variance of injection moulding process parameters and clay particles size effects on impact strength, shrinkage and warpage of polyethylene/kaolin clay composites

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Abstract

A correlation effect between particle size of kaolin clay and injection moulding process parameters on impact strength, shrinkage and warpage of high-density polyethylene/kaolin clay (HDPE/KC) composites was carried out using analysis of variance (ANOVA). Kaolin clay with particle sizes of < 75, 75–106 and 106–150 μm was used. The process parameters that were taken into consideration were injection temperature, packing pressure and packing time. In general, experimental results showed that the impact strength and shrinkage of the HDPE/KC composites clearly depended on the injection temperature. However, no clear dependency of the warpage on the injection temperature was observed. Furthermore, clay particle size showed to have an influence only on the shrinkage of the composites, where smaller clay particle size led to injected composite parts with relatively less shrinkage. ANOVA showed that the effect of injection temperature on shrinkage of composites containing clay particle sizes of < 75 and 106–150 μm was statistically significant ($p = 0.01$ and 0.04, respectively). However, the effect of injection temperature on shrinkage of the composite made with clay having particle sizes of 75–106 μm was not significant ($p = 0.07$). ANOVA also indicated that the injection temperature effect on the impact strength of composites that contain clays with particle sizes of < 75 μm and 75–106 μm was significant ($p = 0.03$ and $p = 0.01$, respectively), whereas the injection temperature effect on the impact strength of the composite containing clay with a particle size of 106–150 μm was not significant ($p = 0.17$). Contrary to shrinkage and impact strength, the effect of the studied parameters on the warpage was not statistically significant, which was in good agreement with the experimental observations.

Keywords: Kaolin clay, High-density polyethylene, Injection moulding, Shrinkage, Warpage

Introduction

Clay particles are well-known fillers, which are widely used in polymer composites due to their outstanding properties [1–3]. The obtained composite materials usually possess both the properties of polymers and clays. Due to their improved properties, they are used in many industrial applications such as automobiles, electrically conductive materials and other applications, where enhanced stiffness, improved strength, high-impact properties and thermally stable materials are needed [4]. Similar to polymers, the manufacturing of industrial parts from polymer composites widely uses the injection moulding process through which the melted polymeric materials are injected into a mould. This process depends on a variety of variables, such as injection temperature, injection pressure and packing time. Although the injection moulding process is considered to be very stable, however, due to some interactions between such parameters, the resultant product's quality could be dramatically affected. Therefore, this process requires optimization, which could improve the physical and mechanical properties of the final products. The impact strength of polymer composites is one of the mechanical properties that might be greatly affected by the processing method used [5–10]. Furthermore, the impact strength of polymer composites is determined by interactions of many factors and parameters; therefore, it may exhibit a complex behaviour under impact loadings [11].

Understanding how injection moulding processing parameters could affect the mechanical properties of the manufactured polymer composites is an important topic, which is under lots of investigation nowadays. Injection temperature, injection pressure and time are probably the most important processing parameters affecting the injection moulding process. These parameters may cause a significant change in the dimensions of the final product after being injected at moulding temperature, resulting in some defects, which cannot be avoided [12–14]. The reduction in volume from the proposed size during the cooling process is one of these defects, which is known as shrinkage [15]. Similarly, during the solidification process, stresses build up which eventually give rise to residual stresses in the finishing product, resulting in what is known as warpage [15–17].

In this work, design of experiment (DOE) was obtained via the Taguchi method [18, 19]. The latter is a method that uses an orthogonal array, which provides as much data with few numbers of specimens using a table that generates a highly effective and efficient number of experimental trials. Analysis of variance (ANOVA) could be performed on the data obtained from the Taguchi design to determine the values of parameters so that the significance of the response and the contribution can be obtained. In recent years, this method has been extensively used for polymer composite materials [20–22]. In this work, the correlation effect of kaolin clay particle size and injection moulding process parameters on impact strength, shrinkage and warpage of high-density polyethylene/kaolin clay (HDPE/KC) composites was investigated. Three different clay particle sizes (< 75, 75–106 and 106–150 μm) and injection process parameters, namely injection temperature, packing pressure and packing time, were examined. The analysis of each factor and its significance to the response and the contribution of each process parameter towards the output characteristic using ANOVA was performed. To the best of our knowledge, this is the first report on the correlation effect between particle size of Libyan kaolin clay and injection moulding process parameters on impact strength, shrinkage and warpage of HDPE/KC composites using ANOVA.

Experimental

Materials

High-density polyethylene (HDPE) was used as the matrix polymer, which was obtained from SABIC Saudi Arabia (HDPE F00952, melt flow index of 0.05 g/10 min and density of 952 g/cm^3). Kaolin clay was supplied by Industrial Research Center, Tripoli, Libya (collected from Sabha city in Libya). It was sieved through different sieve sizes to obtain three different clays with particle sizes of < 75, 75–106 and 106–150 μm.

Composite preparation

Kaolin clay was dried using air circulating oven at 85 °C for 24 h and mixed with the polymer in an ultra-centrifugal mill (ZM 200–RETSCH, Germany) to obtain a very fine powder of the mixture (at 2wt% clay to polymer). The final mixing was then carried out by melt mixing method using a twin-screw extruder (Brabender, Germany) at a screw speed of 35 r.p.m. and L/D ratio of 48 at the temperature range of 160–200 °C. The extruded composites were cooled in air and then ground. Samples for impact strength, shrinkage and warpage measurements were prepared in an injection moulding machine (Xplore 12 ml, the Netherlands) at various injection temperatures, packing pressures and packing times.

Shrinkage measurements

Shrinkage is usually measured as the reduction that occurs in a linear direction when a polymeric object cools down to room temperature after being injected at moulding temperature. Three measurements for each trial were taken, and the shrinkage (*S*) was obtained using the following formula.

$$S = \frac{L_c - L_{\mathrm{ave}}}{L_c}$$

where L_c is the actual mould cavity length (mm), and L_{ave} is the average sample length (mm).

The actual mould cavity length (L_c) is calculated as:

$$L_c = L\ [1 + \alpha\ (T_{\mathrm{mould}} - T_{\mathrm{ambient}})]$$

where α is the coefficient of thermal expansion for steel (6.45×10^{-6} 1/°*F*), L is the measured cavity length (mm), T_{mould} is the mould temperature in °F and T_{ambient} is the ambient temperature in °F.

Warpage measurements

The thickness of the test sample was measured at three different places using a digital micrometre. Three measurements for each trial were taken, and an average warpage value was used. The warpage (*Z*) was calculated using the following formula:

$$\mathrm{Z} = \mathrm{h} - \mathrm{t_a}$$

Table 1 Injection moulding process parameters and their level selection

Parameters (symbols)	Units	Level 1	Level 2	Level 3
Injection temperature (IT)	°C	180	200	220
Packing pressure (PP)	Bar	6	8	10
Packing time (PT)	s	1	2	3

Table 2 Experimental design

Trial no.	IT (A)	PP (B)	PT (C)
1	180	6	1
2	180	8	2
3	180	10	3
4	200	6	2
5	200	8	3
6	200	10	1
7	220	6	3
8	220	8	1
9	220	10	2

where h is the depth of the mould cavity (mm), and t_a is the average test sample thickness (mm).

Impact strength test

The Charpy impact test was carried out to determine the impact strength of all HDPE/KC composites using an impact tester (CEAST Resil, Italy), with an impact energy of 15 J. The specimens for the test were prepared and notched according to the standard method ISO 179. Five specimens were tested for each sample, and an average value was taken.

Experimental design and process parameters

The injection moulding process parameters, which were investigated, are injection temperature, packing pressure and packing time. These parameters or factors were set at three levels for three different composites containing different clay particle sizes (< 75, 75–106 and 106–150 μm) as shown in Table 1. The experimental design was carried out using the statistical Taguchi method by the Minilab-19 software as shown in Table 2.

Results and discussion

Shrinkage, warpage and impact strength

The experimental values of shrinkage, warpage and impact strength of all obtained composites are shown in Table 3. The results are plotted in Fig. 1, which shows the relationship between the shrinkage, warpage and impact strength with the studied process parameters (trials).

The results showed that shrinkage increases as the injection temperature increases for all composites containing clay regardless of its particle size. This is in agreement with a simulation study carried out by Rongji et al. [16] on the shrinkage of plastics. Similarly,

Table 3 Shrinkage, warpage and impact strength of HDPE/KC composites with different clay sizes for each trial, which were obtained experimentally

Clay particle size (μm)	Parameters A:B:C	Shrinkage	STD	Warpage (mm)	STD	Impact strength (kJ/m^2)	STD
< 75	180:6:1	0.021	0.003	0.06	0.01	14.09	3.3
	180:8:2	0.014	0.001	0.04	0.00	13.70	1.5
	180:10:3	0.022	0.001	0.05	0.01	15.69	2.3
	200:6:2	0.032	0.001	0.06	0.03	16.20	2.1
	200:8:3	0.023	0.003	0.07	0.00	13.67	2.3
	200:10:1	0.032	0.005	0.04	0.00	15.23	3.8
	220:6:3	0.032	0.004	0.04	0.00	16.64	1.2
	220:8:1	0.032	0.003	0.04	0.00	21.51	8.8
	220:10:2	0.035	0.001	0.06	0.03	19.05	3.8
75–106	180:6:1	0.050	0.002	0.03	0.02	12.34	0.8
	180:8:2	0.042	0.001	0.04	0.00	12.54	0.9
	180:10:3	0.053	0.003	0.04	0.00	11.37	1.2
	200:6:2	0.061	0.001	0.07	0.00	12.42	0.9
	200:8:3	0.056	0.002	0.05	0.01	13.62	3.5
	200:10:1	0.067	0.004	0.07	0.00	12.50	1.2
	220:6:3	0.059	0.003	0.06	0.01	17.05	1.7
	220:8:1	0.070	0.004	0.06	0.01	16.66	1.7
	220:10:2	0.057	0.001	0.02	0.04	23.15	2.3
106–150	180:6:1	0.051	0.001	0.05	0.01	12.24	2.3
	180:8:2	0.041	0.001	0.04	0.00	15.80	2.7
	180:10:3	0.053	0.003	0.11	0.07	12.00	0.8
	200:6:2	0.063	0.001	0.05	0.01	15.67	1.9
	200:8:3	0.055	0.003	0.05	0.01	14.47	1.6
	200:10:1	0.063	0.004	0.07	0.03	16.01	2.4
	220:6:3	0.056	0.001	0.08	0.03	17.74	4.2
	220:8:1	0.063	0.001	0.06	0.01	14.50	3.2
	220:10:2	0.062	0.003	0.06	0.01	16.19	0.3

the results showed the impact strength value slightly depended on the injection temperature, which was the case for all composites with different clay particle sizes. On the other hand, warpage showed no clear dependence on the process parameters and/or clay particle size. These results indicate that the injection temperature had the highest influence on the shrinkage followed by the impact strength of the composites.

As shown in Fig. 1, one can also see that HDPE/KC composites with relatively smaller clay particle size (i.e. <75 μm) had lower shrinkage values compared to the composite with medium and large particle sizes of 75–106 and 106–150 μm. These results are in good agreement with Kim et al. [23], who showed that there was a decrease in shrinkage when smaller particles of talc were used in poly (butylene terephthalate)/poly (ethylene terephthalate)/talc composites. Figure 1 also shows that there was no clear effect of clay particle size on the warpage and impact strength of the composites.

ANOVA results

ANOVA was carried out to determine which of these process parameter conditions and/or clay particle size effects on the shrinkage, warpage and impact strength were

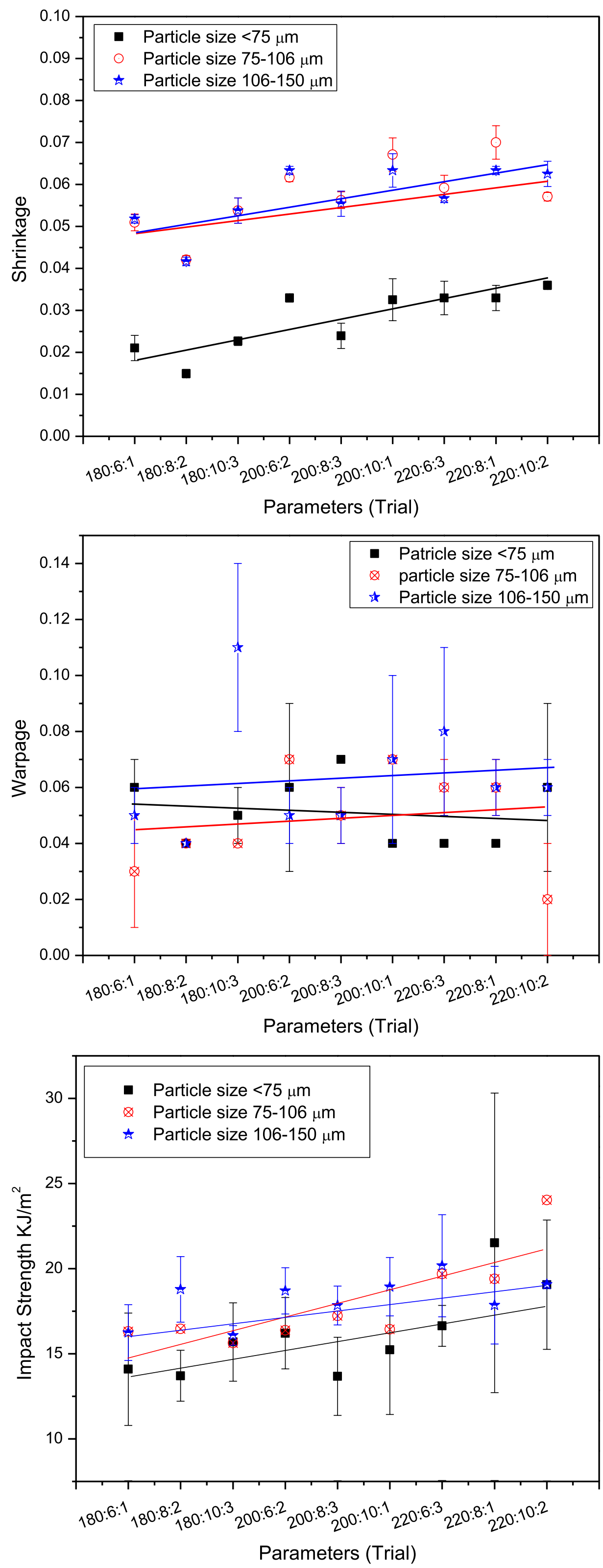

Fig. 1 Correlation effect between shrinkage, warpage and impact strength and process parameters (trial number) for composites containing various clay particle sizes

significant. One-way ANOVA (single factor) was used, and the results are shown in Table 4, which shows the injection temperature effect on shrinkage for HDPE/KC composites with various clay particle sizes. As can be seen, the composites with small and large clay particle sizes (i.e. < 75 and 106–150 μm) showed a p-value < 0.05 which means that the injection temperature effect on the shrinkage was significant. Contrary, for the composite with a medium clay particle size of 75–106 μm, the effect of injection temperature on shrinkage was not significant (p-value > 0.05).

ANOVA was also carried out on other parameters, namely packing pressure and packing time. They all showed a p-value > 0.05, which indicates that the effect of these parameters on shrinkage was not significant (see Additional file 1: Tables S1 and S2 in supporting information). Similarly, the ANOVA results on warpage showed a p-value of > 0.05, which indicates that the studied parameter effect on warpage was not significant.

ANOVA analysis of injection temperature effect on the impact strength of composites with various clay particle sizes of < 75, 75–106 and 106–150 μm are shown in Table 5. The p-value obtained for composites with small and medium clay particle sizes (i.e. < 75 μm and 75–106 μm) were < 0.05, which shows that the injection temperature effect on the impact strength was significant. The p-value for the composite with a relatively large clay particle size of 106–150 μm was > 0.05, which means that the injection temperature effect on the impact strength of this composite was not significant.

Similarly, ANOVA tests were carried out on other parameters, namely: packing pressure and packing time. They all showed a p-value > 0.05, which means that these parameter effects on the impact strength of the composite were not significant (see Additional file 1: Tables S6 and S7). Also, the effect of the same parameters (injection temperature, packing pressure and packing time) on warpage was found to be not significant (they all showed a p-value > 0.05) (see Additional file 1: Tables S3–S5).

Table 4 One-way ANOVA results for shrinkage: effect of injection temperature

	Groups	*Count*	*Sum*		*Average*		*Variance*
< 75 μm	Melt 180	3	0.058		0.019		1.66E−05
	Melt 200	3	0.089		0.029		2.59E−05
	Melt 220	3	0.101		0.033		3.01E−06
75–106 μm	Melt 180	3	0.146		0.048		3.71E−05
	Melt 200	3	0.185		0.061		2.93E−05
	Melt 220	3	0.186		0.062		4.81E−05
106–150 μm	Melt 180	3	0.147		0.049		4.2E−05
	Melt 200	3	0.182		0.060		2.09E−05
	Melt 220	3	0.182		0.060		1.32E−05
	Source of variation	*SS*	*df*	*MS*	*F*	*p-value*	*F crit*
< 75 mm	Between groups	0.0003	2	1.65E−04	10.88	0.0101	5.143
	Within groups	0.0001	6	1.52E−05			
	Total	0.0004	8				
75–106 μm	Between groups	0.0003	2	0.00016	4.39	0.066	5.143
	Within groups	0.0002	6	3.81E−05			
	Total	0.0005	8				
106–150 μm	Between groups	0.0002	2	0.00013	5.39	0.045	5.143
	Within groups	0.0001	6	2.53E−05			
	Total	0.0004	8				

Table 5 One-way ANOVA results of impact strength: effect of injection temperature

	Summary						
	Groups	*Count*		*Sum*		*Average*	*Variance*
< 75 µm	Melt 180	3		43.492		14.497	1.107
	Melt 200	3		45.116		15.038	1.632
	Melt 220	3		57.217		19.072	5.941
75–106 µm	Melt 180	3		36.259		12.086	0.389
	Melt 200	3		38.558		12.852	0.449
	Melt 220	3		56.871		18.957	13.224
106–150 µm	Melt 180	3		40.05		13.35	4.515
	Melt 200	3		46.167		15.389	0.655
	Melt 220	3		48.434		16.144	2.629
	ANOVA						
	Source of variation	*SS*	*df*	*MS*	*F*	*p-value*	*F crit*
< 75 µm	Between groups	37.49	2	18.74	6.47	0.031	5.14
	Within groups	17.36	6	2.89			
	Total	54.85	8				
75–106 µm	Between groups	85.05	2	42.52	9.07	0.015	5.143
	Within groups	28.12	6	4.68			
	Total	113.18	8				
106–150 µm	Between groups	12.53	2	6.26	2.41	0.17	5.14
	Within groups	15.60	6	2.60			
	Total	28.14	8				

Conclusions

A correlation effect between clay particle size and injection moulding process parameters on impact strength, shrinkage and warpage of high-density polyethylene/kaolin clay (HDPE/KC) composites was carried out successfully using analysis of variance (ANOVA). The effect of three different clay particle sizes (< 75, 75–106 and 106–150 µm) and injection process parameters, namely injection temperature, packing pressure and packing time, were examined. The results obtained experimentally showed that the most influencing process parameter regardless of the clay particle size on shrinkage and impact strength was the injection temperature. It was found that the shrinkage was the most influenced by the injection temperature followed by the impact strength, whereas warpage showed no clear dependency on the injection temperature. The shrinkage clearly increased as the injection temperature increased regardless of the clay particle size used. Furthermore, the results indicated that smaller clay particle sizes led to composites with less shrinkage. Warpage on the other hand was not affected by neither clay particle size nor injection moulding process parameters.

ANOVA results showed that the effect of injection temperature on shrinkage of composites containing small and large clay particle sizes (< 75 and 106–150 µm) was statistically significant ($p = 0.01$ and 0.04, respectively). Moreover, the injection temperature effect on shrinkage of the composite containing medium clay particle sizes of 75–106 µm was not significant ($p = 0.07$). Similarly, ANOVA indicated that the injection temperature effect on the impact strength of composites with small and medium clay particle sizes of < 75 µm and 75–106 µm was significant ($p = 0.03$ and 0.01, respectively). However, the injection temperature had no significant effect on the impact strength of the composite with a large clay particle size of 106–150 μm ($p = 0.17$). ANOVA also showed that other process parameters' (i.e. packing pressure and packing time) effect on shrinkage, warpage or impact strength of the composite was not statistically significant, which was in good agreement with the observations obtained experimentally.

Abbreviations

ANOVA	Analysis of variance
DOE	Design of experiment
IT	Injection temperature
HDPE/KC	High-density polyethylene/kaolin clay
HDPE	High-density polyethylene
PP	Packing pressure
PT	Packing time

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s44147-022-00166-5.

Additional file 1: Table S1. One way ANOVA results of Shrinkage: effect of Pack pressure. Table S2. One way ANOVA results of Shrinkage: effect of Pack Time. Table S3. One way ANOVA results of Warpage: effect of Injection temperature. Table S4. One way ANOVA results of Warpage: effect of Pack pressure. Table S5. One way ANOVA results of Warpage: effect of Pack Time. Table S6. One way ANOVA results of Impact strength: effect of Pack pressure. Table S7. One way ANOVA results of Impact strength: effect of Pack Time.

Acknowledgements

The authors would like to thank the Libyan Polymer Research Center.

Authors' contributions

AT performed the preparation of samples and testing. WE assisted in the preparation and testing and was a major contributor in writing the manuscript. HE contributed to the writing and discussion of the results. AK contributed to the writing, discussion of the results and proofreading. The author(s) read and approved the final manuscript.

Funding

The authors declare no funding has been received for the research.

Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author upon reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

Received: 22 September 2022 Accepted: 28 November 2022
Published online: 13 December 2022

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