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Investigation of Mechanical and Melt Flow Properties of Polyamide 12 Composites of Different Reinforcement Materials

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دراسة الخواص الميكانيكية وانسياب المصهور لمركب البولي أميد 12 من مواد مالئة مختلفة

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Abstract:
The influence of inexpensive different fillers: marble powder, Libyan kaolin clay and white cement on the polyamide 12 (PA12) composite, to improve the mechanical properties and reduce the cost was investigated. Composites with (10, 20, 30, 40 and 50 wt% filler content) were produced using injection molding. The mechanical and melt flow properties were investigated. The results showed that the ultimate tensile strength, elongation at break, Young’s modulus, and Shore hardness of PA12 composites with 10 wt % marble and white cement were higher than that of pure PA12. In all PA12 composites, Young’s modulus and Shore hardness were higher than that of pure PA12 and increased with increasing the filler content. Although the impact strength values of PA12 composites decreased with increasing the filler content, the decrease in the impact strength values of PA12 composites with 10 wt % filler content was not significant compared to pure PA12. These results demonstrated that the addition of 10 and 20 wt % of marble and white cement to PA12 appear to improve certain properties such as ultimate tensile strength, elongation at break, Young’s modulus, and hardness with reasonable processability and no significant influence in the impact properties.

Keywords: Polyamide, Filler, Mechanical properties, Melt flow

الملخص
تم دراسة تأثير مواد مالئة مختلفة غير مكلفة بإضافة مسحوق الرخام، طين الكاولين الليبي والأسمنت الأبيض على مركب البولي اميد 12 (PA12)، لتحسين الخواص الميكانيكية وتقليل التكلفة. تم إنتاج المواد المركبة التي تحتوي على (10، 20، 30، 40 و 50٪ بالوزن من مادة المالئة) باستخدام القولبة بالحقن. تم دراسة الخواص الميكانيكية وانسياب المصهور. أظهرت النتائج أن قوة الشد النهائية، والاستطالة عند الكسر، ومعامل يونج، وصلادة شور لمركبات PA12 مع 10٪ بالوزن من الرخام والأسمنت الأبيض كانت أعلى من تلك الخاصة بالبولي اميد PA12 النقي. في جميع مركبات PA12، كان معامل يونج وصلادة شور أعلى من PA12 النقي ويزداد مع زيادة محتوى المادة المالئة. على الرغم من أن قيم مقاومة الصدم لمركبات PA12 انخفضت مع زيادة محتوى المادة المالئة، إلا أن الانخفاض في قيم مقاومة الصدم لمركبات PA12 التي تحتوي على 10٪ بالوزن من محتوى المالئة لم يكن كبيرًا مقارنة بالبولي اميد PA12 النقي. أظهرت هذه النتائج أن إضافة 10 و20% بالوزن من الرخام والأسمنت الأبيض إلى البولي اميد PA12 قد ادت الى تحسين خصائص معينة مثل قوة الشد القصوى، والاستطالة عند الكسر، ومعامل يونج، والصلادة مع قابلية اعادة القولبة وليس لها تأثير ملحوظ على خاصية مقاومة الصدم.

الكلمات المفتاحية: بولي أميد، مادة مالئة، الخواص الميكانيكية، انسياب المصهور.

Introduction

Polyamides are thermoplastic polymers which have repeated amide groups in the chain structure. They are classified as engineering thermoplastics and have a wide spectrum of properties, including moderate mechanical and excellent wear-resistant properties [1-2]. Due to their excellent properties, polyamides are extensively employed in consumer products, medicine, aircraft, aerospace, automotive and other industries. Polyamides are the materials of choice for designing and manufacturing final production parts through selective laser sintering (SLS) and injection molding [3]. SLS is a typical rapid prototyping technology employed with polymer powders to fabricate products layer by layer with various properties that are comparable to those obtained by injection molding. In point of fact, the SLS process offers several advantages over conventional production techniques such as injection molding. These advantages are short design to manufacturing cycle time, high geometrical freedom and customized components.

One of the main disadvantages of the PA12 processed by SLS is high porosity, and low molecular weight, which deteriorate its mechanical properties [4-5]. Moreover, the cost of PA12 powder is very high; even simple sintered parts with volumes of 10 cm^3 cost thousands of dollars for the raw material alone and the use of PA12 powders often creates unsatisfactory mechanical properties, notch sensitivity and high-water absorption [2,6,7]. These disadvantages of SLS and PA12 could be improved by using injection molding and the addition of fillers and additives. Fillers are added to the polymers in commercial production primarily for reasons related to cost reduction and properties improvement. Fillers has been extensively used with engineering polymers to obtain a desirable combination of properties such as improved tensile strength, impact strength, and heat distortion temperature together with ease of flow.

Fillers such as carbon nano-tubes [8], graphite oxide [9], silicon dioxide [10] metal particles [11], cement [12], clay [13] and others have been added to reinforce the PA12 in manufacturing process. Although there are many attempts to use of different types of fillers to reinforce the PA12, there is still an enormous interest to use other fillers to develop PA12 composites with enhanced properties. Therefore, the aim of the work presented in this paper is to explore the influence of different types of fillers including; marble waste powder, Libyan kaolin clay and white cement, for the purpose of improving the mechanical properties and reducing the cost of PA12 products using injection molding process.

Material and methods

In this work, PA12 and different fillers (marble waste powder, Libyan kaolin clay and white cement) were used. PA12 powder (PA1550), which is an ultrafine powder of polyamide with a narrow particle size and nearly round particle shape was supplied by EXCELTEC. Marble powder with average particles size 30-150µm, which is a waste product from the marble cutting industry. Libyan kaolin clay from southern Libya (collected from Jarmah Member, Sabha city in Libya) which consists of very small particles of alumina and silica. It was grinding manually and passed through a mesh 30-150 µm. The main chemical composition and general properties of Libyan kaolin were reported by Shiwa and Hussin [14]. According to Eshmaiel [15] Libyan kaolin clay is relatively high in silica and its physical properties could be referenced as ASTM C618 class. White cement with average size 30-150 µm was obtained from domestic cement industries.

Preparation of composites

The PA12 was mixed with each material (marble waste powder, Libyan clay and white cement) in different proportions by weight, 90:10, 80:20, 70:30 60:40 and 50:50 manually for 10 to 15 min to obtain homogenous mixture. An injection molding machine (Xplore 12ml) was used to homogenized the mixture again and to prepare the specimens for mechanical tests. The operating pressure and temperature of the injection molding machine was1.6 MPa and 200°C respectively, and the processing time for each sample was 1min approximately. Details of the composites and codes are reported in Table 1.

Characterization

Mechanical properties

The specimens for tensile strength were shaped according to ISO (527-2-5A), the dimensions of the specimens were 75 × 12.5 × 2 mm. Tensile strength tests were carried out using a single column tensile tester from (AML instruments) with a load range of 0 to 2400N, accuracy +/- 0.5% and test speed was 5mm/min. Four specimens were tested for PA12 and each composite.

The Charpy impact test was carried out to determine the impact strength of the PA12, and all composite materials using (CEAST Resil Impactor tester), with an impact energy of 15 J. Impact specimens were shaped according to ISO 179-180with the dimensions of90 × 4 × 10mm. Four specimens were tested for PA12 and each composite.

Shore Dhardnesstest was performed using the shore durometer (RayRan). The specimens were prepared according to ISO 868. Ten measurements were taken for PA12 and each composite.

Density

The density of PA12 and composite specimens was determined according to the ISO 1183-3immersion method.

Melt flow rate and viscosity

Examples of desirable melt flow properties may include melt flow rate (MFR)and viscosity. Thus, MFR and viscosity of the PA12 and composites were studied using CEAST modular line melt flow models 7024 according to ISO 1133. The tests were carried out at 235 °C at 5.0 kg.

Table 1 Composites composition and codes.

Nº	Composite code	PA12, wt%	Marble, wt%	Libyan clay, wt%	white cement, wt%
1.	PA12	100	0	0	0
2.	PAM10	90	10	0	0
3.	PAM20	80	20	0	0
4.	PAM30	70	30	0	0
5.	PAM40	60	40	0	0
6.	PAM50	50	50	0	0
7.	PALC10	90	0	10	0
8.	PALC20	80	0	20	0
9.	PALC30	70	0	30	0
10.	PALC40	60	0	40	0
11.	PALC50	50	0	50	0
12.	PAWC10	90	0	0	10
13.	PAWC20	80	0	0	20
14.	PAWC30	70	0	0	30
16.	PAWC40	60	0	0	40
17.	PAWC50	50	0	0	50

Results and discussion

Mechanical properties

Table 2, illustrates the ultimate tensile strength (UTS), elongation at break (EAB), Young's modulus, impact strength and shore hardness for pure PA12 and all composites. Values between brackets are standard deviation (STD).

Looking closer at the results, UTS of PA12 composites with 10% marble and white cement were relatively higher than that of pure PA12.The highest value of UTS was observed by PAWC10 (PA12 composite with 10%white cement). The decrease in the UTS of PA12 after adding 10% Libyan kaolin clay was relatively small. Increasing the content of these fillers to PA12 matrix caused decrease in the UTS. This may indicate that these fillers (marble, Libyan clay, and white cement) could enhance the UTS to some extent and then do the opposite as their content was increased. Similar findings were indicated by Aldahsh [16]. He found that, when wt% of cement is < 10%, the UTS of the composite increases steadily, and then decreases gradually with increasing the cement content. He also found that the addition of cement particles to PA12 diminishes the elongation of the composite. This can be in line with the results of this study. However, the highest values of EAB were observed by PAWC10 (with 10%white cement), PAM10 (with 10%marble) and PALC10 (with 10% Libyan clay), respectively. The EAB values of PAWC10 (with 10%white cement) and PAM10 (with 10%marble) were higher than that of pure PA12. The same trend was observed for the EAB results, as in the case of UTS results. Increasing the content of these fillers to PA12 matrix caused decrease in the EAB. It is to be noted that the mechanical properties of a polymer composites can be strongly depend on factors, such as the particle size of the filler, the interface adhesion between the filler and the matrix, and the filler loading [17].

Young's modulus values increased as expected after the addition of the solid fillers. This is because Young's modulus is markedly improved by adding rigid filler particles to a polymer matrix, since the rigidity of fillers is generally higher than that of the polymers. Consequently, the composite modulus consistently increases with increasing filler content [18]. The enhancement in the Young's modulus may results improvement in the mechanical stability of PA12 composites in comparison to pure PA12, as reported in the literature [19]. Therefore, it is important to declare that obtaining high Young's modulus is an effective strategy to expand the lifetime of polymeric products.

Table 2 Mechanical properties of pure PA12 and PA12 composites.

Nº	Composite code	Ultimate tensile strength, MPa	Elongation at break, %	Young's modulus, MPa	Impact strength, $KJ.m^{-2}$	Shore hardness
1.	PA12	47.9 (2.1)	75.4 (3.2)	695.9 (33.1)	4.4 (0.05)	71.0 (0.1)
2.	PAM10	49.4 (3.7)	86.4 (3.0)	719.7 (31.3)	4.0 (0.2)	71.1 (0.3)
3.	PAM20	47.5 (2.6)	33.7 (2.8)	962.6 (52.6)	3.9 (0.1)	72.4 (0.1)
4.	PAM30	47.9 (5.4)	19.7 (2.1)	1140.7 (80.2)	3.1 (0.1)	75.0 (1.1)
5.	PAM40	42.0 (1.3)	10.9 (1.9)	1252.5 (51.5)	2.8 (0.3)	76.3 (1.1)
6.	PAM50	42.4 (3.8)	05.8 (2.5)	1729.5 (79.4)	2.5 (0.3)	77.1 (1.1)
7.	PALC10	47.4 (8.1)	66.4 (2.4)	1078.5 (55.3)	4.0 (0.1)	71.3 (0.7)
8.	PALC20	46.5 (2.6)	28.0 (3.1)	1347.6 (62.8)	3.9 (0.1)	71.6 (0.2)
9.	PALC30	44.8 (6.8)	08.3 (2.1)	1416.4 (60.9)	2.8 (0.2)	72.0 (0.9)
10.	PALC40	39.0 (4.2)	05.3 (1.8)	1577.1 (58.4)	2.5 (0.1)	73.1 (0.8)
11.	PALC50	48.1 (3.9)	04.3 (1.9)	1899.0 (58.7)	2.2 (0.3)	74.3 (1.2)
12.	PAWC10	49.7 (5.7)	93.1 (1.8)	966.3 (61.9)	4.2 (0.3)	73.3 (0.5)
13.	PAWC20	47.1 (6.6)	47.0 (2.1)	1149.7 (66.0)	3.8 (0.3)	74.0 (1.1)
14.	PAWC30	46.9 (7.1)	24.6 (2.0)	1170.8 (81.2)	3.2 (0.2)	74.7 (0.2)
16.	PAWC40	45.66 (3.2)	11.5 (2.6)	1252.5 (54.4)	3.0 (0.3)	76.5 (0.8)
17.	PAWC50	45.37 (6.3)	08.3 (3.1)	1455.8 (56.8)	2.6 (0.3)	77.5 (0.6)

Impact strength was decreased with the addition of fillers to PA12, as shown in Table 2 and Figure 1. Similar to UTS, EAB and Young's modulus, the impact strength values were decreased with increasing the filler content. Moreover, composites with 10 wt% filler content had higher impact strength than other composites. The maximum impact strength value was observed byPAWC10 (with 10 wt% white cement). The decrease in impact strength could be attributed to the agglomeration of filler's particles. In fact, fillers tend to agglomerate and to resist dispersion as the filler content increases. These usually weaken the interfacial adhesion between filler and polymer and become potential sites for crack growth because of inability of filler to support stress transfer to the polymer matrix [16, 20].

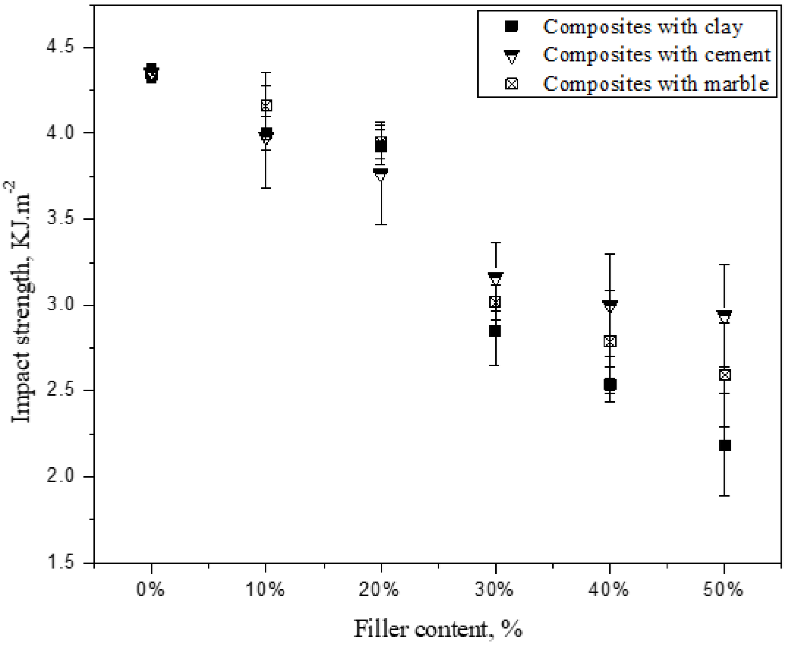

Figure 1: Impact strength versus filler content of different PA12 composites.

As can be seen in Table 2 and Figure 2, the addition of fillers to PA12 provided an increase in the Shore hardness. All composites exhibited higher Shore hardness than PA12.Thiscan be attributed to the high hardness of the filler's particles. According to Marset et al. [21], the addition of inorganic filler into polyamide matrix may led to increase in the hardness, which can be directly related to the improvement in stiffness provided by the addition of inorganic filler. The Shore hardness values were increased with increasing the fillers content. Composites with 50 wt% filler content had higher hardness than other composites. The maximum hardness value was observed byPAWC50 (with 50%white cement).

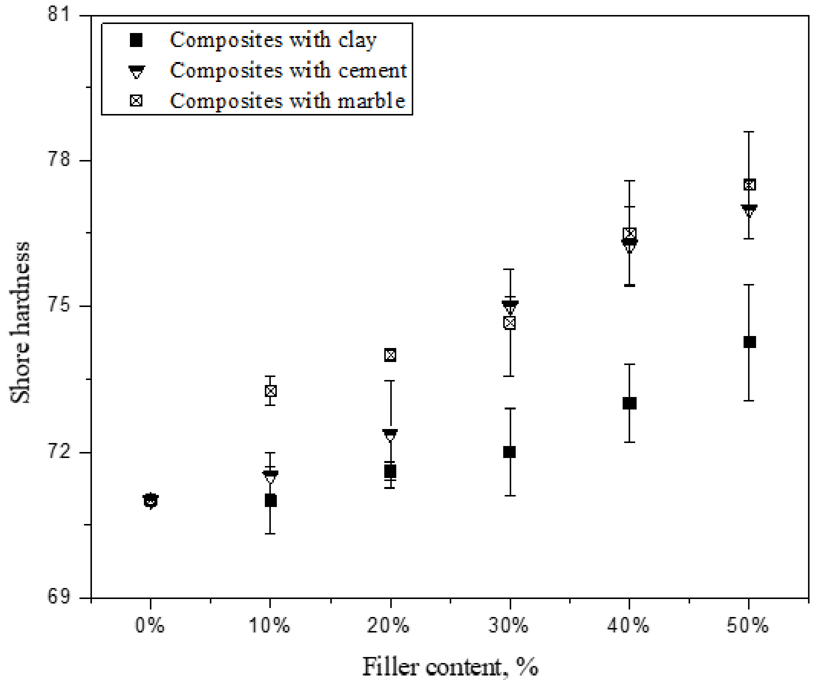

Figure 2: Shore hardness versus filler conten of diffferent PA12 composites.

The improvement of mechanical properties of PA12 composites appears to be dependent on the interaction between the PA12 matrix and filler, dispersion of filler particles into the PA12 matrix, type of filler and filler content. In general, the mechanical properties [2] 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 [22]. As a result of this study, the improvements in the mechanical properties of the PA12 composite were in the following order: PA12 with white cement > PA12 with marble > PA12 with Libyan clay.

Density results

Figure3show that the addition of different fillers with different proportions to the PA 12 matrix affected the density of the PA12. The densities of PA12 composites fabricated using injection molding increased with increasing the filler content in all the composites.

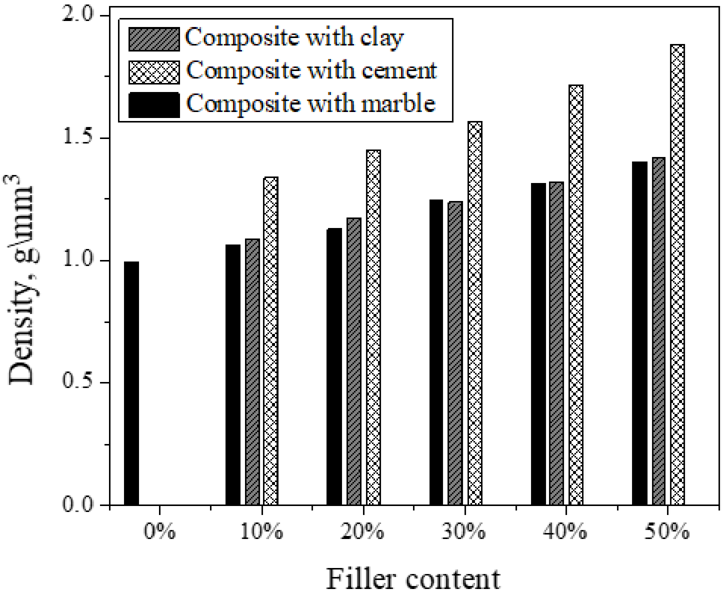

Figure 3: Density results for PA12 and PA12 composites.

The density of pure PA12 was lower than the densities of all the PA12 composites. This was expected because the addition of filler could increase the overall composite density [18]. It is important to declare that the density of PA12 composites with white cement was higher than other composites.

Melt flow rate and melt viscosity results

Table 3, illustrates the MFR and viscosity results, respectively, of PA12 and composites, which contain 10 and 20 wt% filler content. It is important to underline that the addition of different types of filler resulted cleardecrease in MFR and a noticeable increase in the melt viscosity. Increasing the filler content from 10 to 20 wt% resulted more decrease in the MFR and increase in the melt visocity.Simlar finding was reported elsewhere [23]. Nevertheless, the increase in the melt viscosity can cause decrease in the MFR of the polyamide/cement composites [16]. However, the increase in the filler can lead to a restriction of the molecular motion in the composites [24-25]. According to Mohamad et al. [26], the addition of fillers tends to impose extra resistance to flow due to higher restriction to molecular motion of the macromolecules; hence higher viscosity. It was found [27] that the addition of filler may cause restriction to the molecular motion of polymer, which tend to impose extra resistance to flow. It is well-known in literature [28], that the lower the melt flow, the higher the melt viscosity of the polymer. For composites with higher filler contents (30 to 50 wt%), the melt viscosity of each composite was too high to be measured in the above-mentioned conditions.

Table 3 MFR and melt viscosity at different additive content.

Composite code	MFR, g/10 min	Viscosity, Pa.S
PA12	21.26	968.97
PAM10	14.02	1545.41
PAM20	12.01	1933.38
PALC10	12.01	1871.64
PALC20	09.59	2611.93
PAWC10	13.62	1663.78
PAWC20	12.41	2040.47

Conclusion

Composites of PA12 with marble, Libyan clay and white cement were prepared successfully in diffrenet compositions. Mehacincal (UTS, EAB, Young's modulus, impact strength, and Shore hardness) and processing (MFR, melt viscosity and density) properties were performed to study the influence of these fillers on PA12. The following conclusions can be drawn in the light of above results:

- The improvements in the mechanical properties of the PA12 composites were in the following order: PA12 with white cement > PA12 with marble > PA12 with Libyan clay.
- The mechanical properties such as UTS, EAB, Young's modulus, and Shore hardness of PA12 composites with 10% marble and white cement were higher than that of pure PA12.
- Young's modulus and Shore hardness of PA12 composites were higher than that of pure PA12 and increased with increasing the filler content.
- Although the impact strength values of PA12 composites were decreased with increasing the filler content, the decrease in the impact strength values of PA12 composites with 10% filler content was not significant in comparison to pure PA12.
- The densities of PA12 composites were higher than that of pure PA12 and increased with increasing the filler content.
- The addition of these fillers resulted in an apparent decrease in MFR and a noticeable increase in the melt viscosity of PA12. MFR and melt viscosities of PA12 composites with 30 to 50% filler content are too high to be measured in the test conditions.

It should be concluded that the addition of 10 to 20 % of fillers such as marble and white cement to PA12 appears to reduce the cost and improve certain properties such as UTS, EAB, Young's modulus, and hardness with reasonable processability and no significant influence in the impact properties.

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