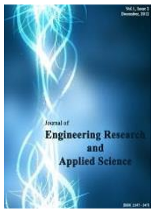
Journal of Engineering Research and Applied Science
Available at www.journaleras.com
Volume 7 (2), December 2018, pp 937-943
ISSN 2147-3471 © 2018



Influence of the addition chestnut shell to kaolin/polyester composites

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Accepted 29 October 2018

Abstract
In this work, the fiber of the chestnut shells from the industrial food wastes, and 2 different kinds of kaolin were evaluated to reinforcement in the polyester composite to enhance the mechanical properties. The chestnut shell fibers were added to the polymer matrix during the composite manufacture with an increasing proportion from 3 -10 in volume% and kaolin standard loading as 3% was used. And also the control samples were produced only with chestnut shell fiber/polyester. The polymer composites were characterized by scanning electron microscope, thermogravimetry analysis. The results showed that the addition of increasing amount of chestnut shells fiber and kaolin affected the mechanical properties of the polymer composites. Generally, the flexural modulus, water absorption, and open porosities increased proportionately with the fiber quantity, whereas flexural strength, thermal stability properties changed inversely proportional to the chestnut shells fiber content.

Keywords: Chestnut shell waste; kaolin; polyester composite; thermo-mechanical properties; characterization.

1. Introduction

In the globalized world, with the impact of industrialization and urbanization, the unconscious use of natural resources due to the emergence of uncontrolled waste which especially in food waste resulted [1]. For this reason, the collection of waste and the use of it as a raw or an ingredient material in the production of new products are important for human health, environmental pollution and national economy [2]. The production of composite materials is increasing day by day in order to evaluate existing resources more efficiently and to meet the new needs that are emerging over time with reasonable costs [3]. The development of environmentally friendly, sustainable materials and corresponding composite materials has become the focus of research today [4]. Natural fibers have been used as reinforcing materials for many years because of its being low cost, having high strength and mechanical properties. For this purpose in the composite industry, non-wood forest products such as chestnut shell, pine cone and tree bark are also evaluated [5].

Many studies had been carried out on the utilization of natural fillers and fibers. Underutilized agro-waste are most importantly rich resources of lignocelluloses materials, some typical example are millet, rice, wheat, corn straw, cocoa husk, corncobs and fiber [6]. The chestnut fillers can be obtained directly from the natural resource; it is cheap and also has advantages due to its renewable nature, and easy availability [7]. Chestnut wastes are usually used as biomass/biochar source. The dimensional stability and mechanical properties of the polypropylene composites filled with chestnut shell flour have been determined by Kaymakci and Ayrilmis. This study has shown that maleate polypropylene treated composites can be used efficiently as the floor covering products due to their high dimensional stability and satisfactory mechanical properties [8].

Kaolinite ($Al_4(OH)_8Si_4O_{10}$) is a clay mineral which is a major component of the industrial clay kaolin [9]. It is a material group which used in ceramic, casting, food, petroleum, filler, paper, plastic, medicine fields. Kaolin is type of clay derived from granite rocks. The production of clay-polymer nanocomposites is an active area of research which benefits from the high surface area of nanoclay particles to greatly improve the thermo mechanical properties of the polymer matrix, even with low filler contents [10]. Leong et al. reported in their work, three types of mineral filler (talc, calcium carbonate and kaolin) were separately mixed with polypropylene. The composites were injection molded and the effects of filler loading on mechanical, flow and thermal properties for different types of filled composites were investigated. The results show that the talc-filled polypropylene composites have the strongest filler-matrix

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interactions, whereas kaolin or calcium carbonate filled polypropylene composite have not good enough interactions like talc-filled polypropylene composites [11]. Al-Asade and Al- Al-Asade and Al-Murshdy studied the addition of kaolin into the unsaturated polyester matrix to prepare particulate composites. Addition of kaolin to an unsaturated polyester resin in the 3-9% shows that kaolin act as a binder and as particulate reinforcement results in improvement in the mechanical properties of the unsaturated polyester via the resulting composite [12]. Another study about kaolin polyester composite was investigated by Ahmed et.al.. In this study, polymeric composite produced from unsaturated polyester based on PET waste with diethylene glycol and untreated or treated kaolin was tested. Thermal and chemical methods for treatment of kaolin were performed. These treatments affected the mechanical and electrical properties of the kaolin-filled polymer composites [13].

The waste chestnut shell and kaolin, together have not been ago studied as reinforcing filler in the production of polyester composite material. In this study, composites were manufactured with using of waste chestnut shell fiber, the different kinds of kaolin and polyester for matrix. The effect of the chestnut shell fiber/kaolin ratios and kind of kaolin was investigated with various tests. The thermal stability of the blends of chestnut shell fiber/kaolin polyester composites was studied with Thermal Gravimetric Analyzer.

2. Material and methods

2.1. Materials

The polyester resin (Polipol 383-G) was used as the matrix that was obtained from the Poliya Composite Resins and Polymers Inc. The mixing ratio used was 100g of polyester resin with 0.625ml accelerator (2% Cobalt naphthenate solution), 3ml hardener (Methyl Ethyl Ketone peroxide, MEKP, Akzo Nobel Products) and 0.5ml Polyethylene graft maleic anhydrite (Sigma Aldrich) as coupling agent. The filling material of discarded chestnut shells was obtained from the chestnut candy company in Bursa/Turkey. Two kind of the kaolin was added as reinforcement materials with chemical composition show in Table (1). This table shows the result of XRF analysis of kaolin. The samples were placed in the XRF machine for elemental analysis. The machine (XRF PANalytical, AXiosmAX minerals) was operated at 50 KV.
The theoretical densities and volumes belonging to the ground chestnut shell, kaolin were determined by using a Micromeritics brand AccuPyc II 1340 model Helium-gas pycnometer (Table 2).

2.2. Preparation of composites

The three fillers i.e., Chestnut shell flour (C), White kaolin (W), Grey kaolin (G) in the powder forms were properly mixed according to the each experiment run given in Table 3. After proper mixing of these fillers, six compositions of hybrid fillers were prepared.

Table 1. Chemical composition of kaolin.

Oxides	Grey kaolin	White kaolin	Oxides	Grey kaolin	White kaolin
Na_2O	0.039		Cr_2O_3	0.137	
MgO	0.196	0.236	Fe_2O_3	4.340	2.326
Al_2O_3	20.259	44.294	NiO	0.038	0.014
SiO_2	59.077	44.749	CuO	0.050	
P_2O_5	2.014	2.770	ZnO	0.009	0.039
SO_3	0.157	0.057	SrO	0.106	0.065
K_2O	6.545	3.420	ZrO_2	0.195	0.088
CaO	1.722	0.759	PbO	0.042	0.274
TiO_2	5.074	0.691			

Table 2. Filling materials theoretical density and volume.

Sample	Mean Density (g/cm^3)	Mean Volume (cm^3)
Chestnut shell	1.4714± 0.0018	1.3547± 0.0016
White kaolin	2.5999± 0.0040	1.0211± 0.0016
Grey kaolin	2.5008± 0.0013	2.3833± 0.0013

Table 3. Mixture volume proportion of composites.

Filler/ Polyester	Composite codes and ratio by volume					
	C1P	C2P	C3P	C4P	C5P	C6P
C^{a*}	3	4	6	7	9	10
P^{b*}	97	96	94	93	91	90
	C1WP	C2WP	C3WP	C4P	C5WP	C6WP
C	3	4	6	7	9	10
W^{c*}	3	3	3	3	3	3
P	94	93	91	90	88	87
	C1GP	C2GP	C3GP	C4GP	C5GP	C6GP
C	3	4	6	7	9	10
G^{d*}	3	3	3	3	3	3
P	94	93	91	90	88	87

a*C: Chestnut shell flour; b*P: Polyester; c*W: White kaolin; d*G: Grey kaolin

Figure 1.shows the whole process of the experimental composite specimen preparation. The predetermined amounts of filler, polyester were mixed together in a suitable beaker with a magnetic stirrer. When the process was finished, all agents, including the hardener, accelerator and coupling agent, with a determined ratio, were added to the previous filler/polyester mixture.

An aluminum mold (Figure 1) of the required dimensions (10mm x 4 mm x 100 mm) was used for the making of samples. The mold was covered with a mold-releasing agent so the samples could be easily removed. The completed mixture was poured into the mold [14].

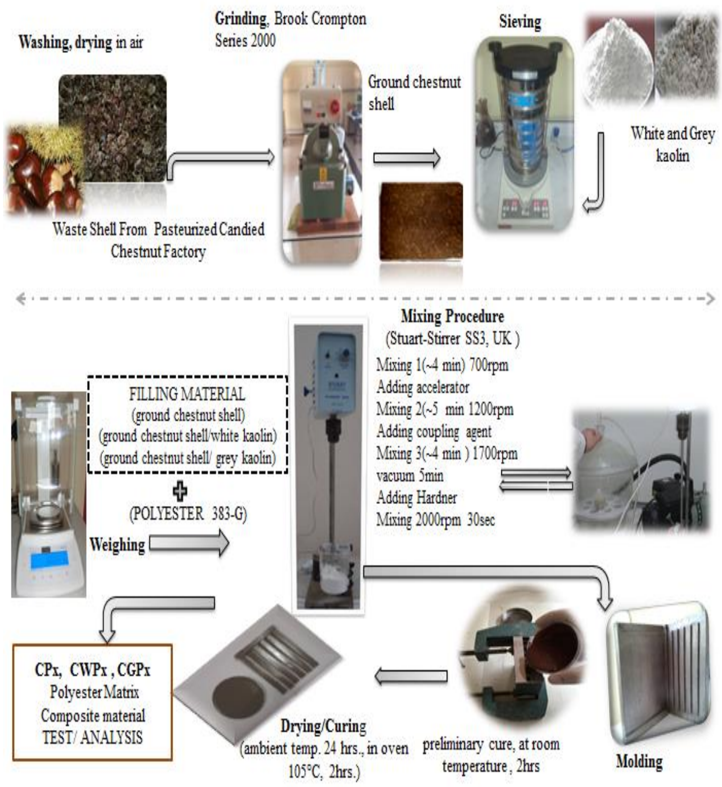

Figure 1. Graphical illustrations for manufacturing process of polyester composites.

2.3.Experimental studies

The flexural properties of the material were determined by 3 point bending test (Fig. 2). The test specimens were with dimensions 10 x 4 x 100 mm. A test speed of 2mm min-1 and Shimadzu AG IC 100 kN test device was used. Results were gathered compliant to the equations given below, by calculating the mean of the five samples.

Figure 2.Applicationof the three point bending test.

Flexural strength (N/mm^2)	=	$(3LP)/(2bd^2)$	E1
Flexural modulus (MPa)	=	$(L^3m)/(4bd^3)$	E2

Where L is the support span (mm), P is the maximum load (N), b is the width of the composite sample (mm), d is the thickness of composite sample (mm), m is the slope of the initial straight line portion of the load–displacement curve.

The indentation hardness of the material was measured by Shore D. The izod impact test was conducted with the device DVT CD, Devotrans Quality Control Test Instruments Ltd., Turkey.

The densities of the composite materials were determined by weighting method. A kit of analytical scale and a weighting jig was used for weighting a specimen of the material in air and fluid. The density is determined by equation: water absorption, bulk density and % open porosity values were calculated.

A, %	=	$(W_D - W_K) / W_K * 100$	E3
B	=	$[W_K / (W_D - W_A)] * \rho_w$	E4
P, %	=	$[(W_D - W_K)/(W_D - W_A)]*100$	E5

Here A is Water absorption, B is Bulk density, P is Apparent porosity W_K is dry weight, W_D is specimen weight in air, W_A is specimen weight in fluid, and ρv is density of the fluid.

Scanning Electron Microscope (SEM., Zeiss Supra 40VP, Germany) was used to obtain microstructural images which were taken from a cracked surface of composites.

Thermal behaviors of the composite samples were investigated using Seteram Lab Sys Evo analyzer. 7–11 mg of accurately measured powdered composite of specimen was kept in alumina crucible. The process is done under nitrogen atmosphere with a flow rate of 20 ml/min by raising its temperature from 20° C to 800° C in steps of 20° C/min.

3.Results and discussion

Table 4 shows the variation in flexural strength of composites with increase in volume fraction of chestnut shell flour. The flexural strengths of chestnut shell-filled composites decreased with increase infiller volume fraction [8, 15]. Similar results were also obtained with the use of palm kernel shell, sugarcane bagasse and pineapple chaffs hybrid polyester composites by Adeosun et al. (2016). Addition of kaolin to filler content influenced to flexural strength as reinforced [16]. The flexural modulus of chestnut shell-filled composites increased with increasing of chestnut shell flour.

In Table 4, the hardness was decreased with an increase in Chestnut shell flour amount for all polyester composites but the addition of kaolin fillers were improved hardness of composites. We believed that it must have been stronger matrix/filler interfacial bond.

The CP composite series has the highest percentage of water absorption in other composite samples. The percentage of water absorption in composite samples increased with the amount of cellulose in the chestnut shell and the percentage of open porosity (Table 4).

Table 4.The effect of chestnut shell flour content on bending and the dimensional stability properties for CP, CWP and CGP composites.

Composite Samples	Flexural Strength (N/mm^2)	Flexural modulus, (MPa)	Hardness, Shore D	Water absorption, (%)	Open porosity,(%)	Bulk Density
C1P	55.21	4,356.82	85.25	0.805	0.955	1.187
C2P	53.38	4,430.69	85.75	0.915	1.088	1.189
C3P	51.81	4,653.77	85	0.921	1.105	1.190
C4P	51.78	4,848.32	85.75	1.038	1.239	1.193
C5P	49.07	5,113.52	86.25	1.212	1.447	1.195
C6P	49.30	5,119.64	85.75	1.273	1.512	1.190
C1WP	62.19	4,132.19	86.000	0.707	0.876	1.240
C2WP	57.10	4,371.91	86.750	0.847	1.053	1.243
C3WP	54.22	4,791.48	87.750	1.005	1.228	1.235
C4WP	53.79	5,025.93	88.000	1.019	1.264	1.240
C5WP	53.51	5,002.15	87.750	1.037	1.287	1.240
C6WP	52.61	5,097.28	88.250	1.128	1.385	1.228
C1GP	55.57	4,630.31	87.250	0.797	0.986	1.237
C2GP	54.98	4,969.10	87.500	0.807	1.075	1.237
C3GP	54.71	5,323.13	87.750	1.135	1.135	1.244
C4GP	53.46	5,325.22	88.500	1.136	1.407	1.238
C5GP	51.06	5,525.18	87.750	1.166	1.439	1.234
C6GP	50.62	5,474.99	88.750	1.171	1.446	1.235

The kaolin clay has reduced the amount of open porosity and improves the coalescence of the matrix and fillings as seen in SEM images. This is supported by Adeosun et al.The scanning electron microscopy (SEM) images of chestnut shell flour, the two types kaolin powders as fillers and fractured surfaces of different composites are shown in Figure 5. Electron micrographs of the structure of kaolin filler (White and Grey) should help illustrate the basic idea contains well developed books of pseudo-hexagonal platy kaolin cluster. When the chestnut shell fiber content is increased to 10vol% the bonding characteristics gets distorted due to insufficient matrix contact with the fiber and the formation of voids leading to reduced mechanical properties [17].

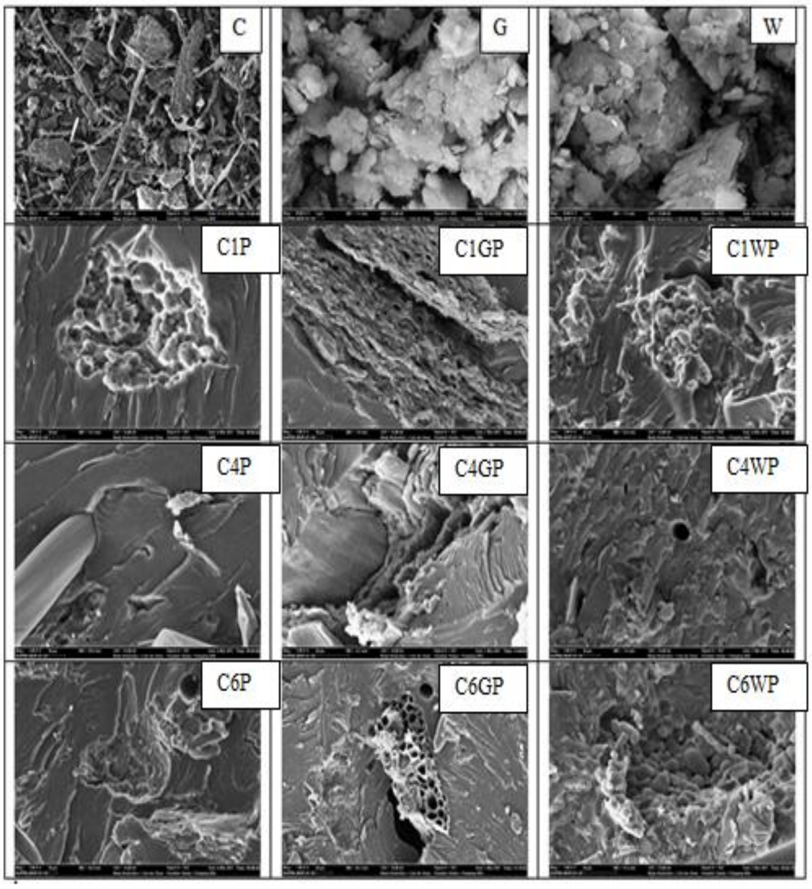

Figure 5.SEM images showing the morphology of fillers (Chestnut shell flour, grey kaolin, white kaolin), polyester composite samples.

Thermal stability results of composite samples by TG, DTG and DTA analysis are presented in Figure 6. The top one is the TG, the bottom one is the DTA and the middle one is the DTG curve of composite samples.

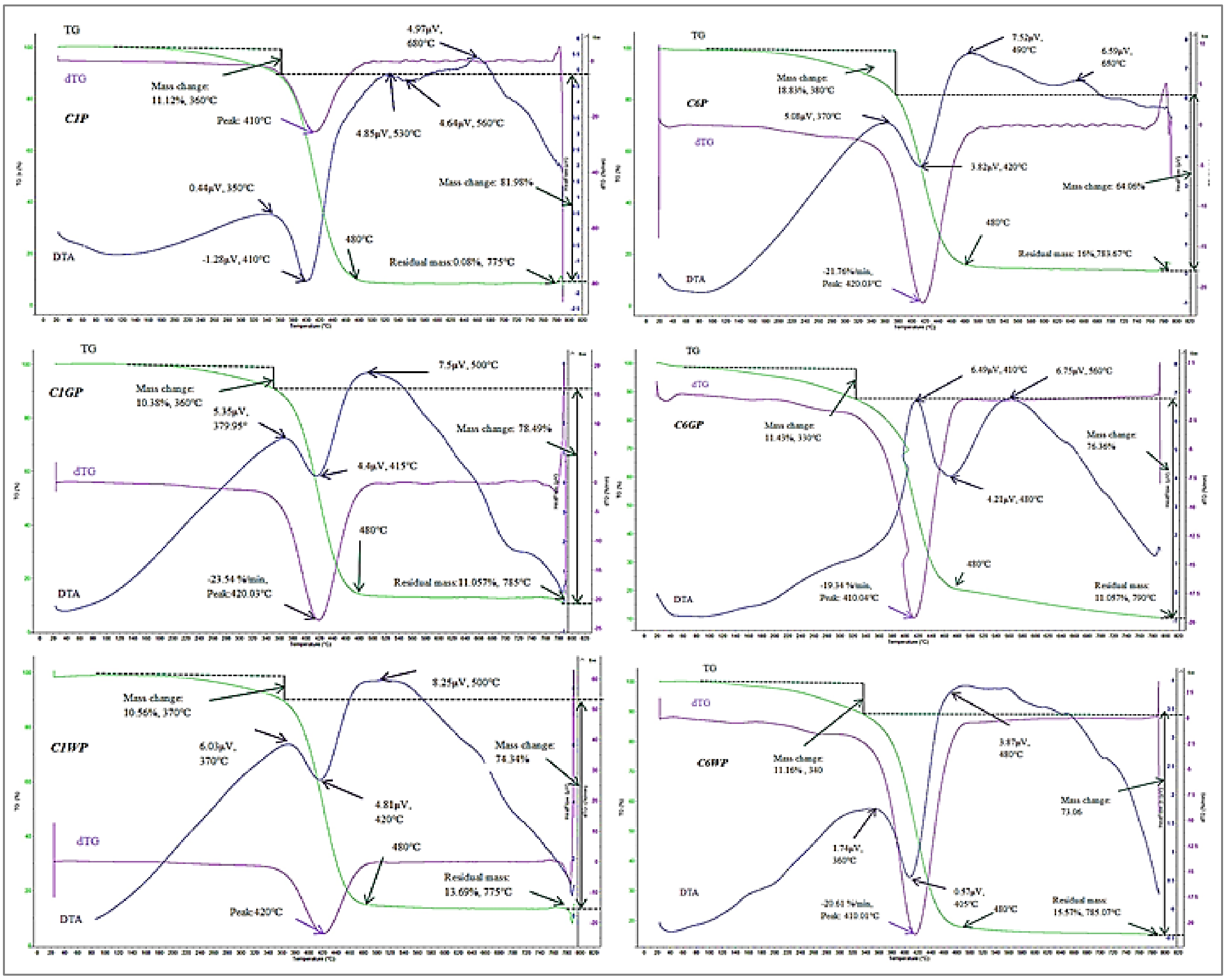

Figure 6. TG, DTG and DTA curves of composite samples.

Minor weight loss occurred up to 100°C-140°C in all composites, which is due to the evaporation of water absorbed in pores and on the surfaces. The lighter substances are removed initially and then heavier materials are removed. The TG curve shows that major degradation occurs in one stage. Thermal stability of the composites are ensured up to 330°C - 360°C and chestnut shell and kaolin burn out occurred at around 480°C which is reasonably a high temperature for polymer composite processing and intended composite applications. DTA curves of composites shows endothermic.

4. Conclusions

In the present work, the discarded, chestnut shell is tested for its potentiality as reinforcement for polymer composite. The influence of fiber content on the mechanical properties, such as flexural strength, impact strength and hardness are significant decreases with fiber content up and with addition of kaolin as reinforced due to improper to chestnut shell polyester composite mechanical features. Chestnut shell and kaolin with together as a filler has been able to withstand sufficient temperature without any serious issues enables it to be used as a potential reinforcement for polymer composites. It is expected that the present behavior may be improved more by changing manufacturing type as use extruder and with injection molding.

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