

Thermal Degradation Kinetics of Polyurethane/Magnetic Activated Carbon Derived from Orange-Peel/Fe₃O₄ Biocomposites

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Abstract

In present work, orange peel derived activated carbon (OPAC) /Fe₃O₄ loaded into polyurethane biocomposites were synthesized with different weight fractions of (OPAC)/Fe₃O₄ via, 0.5, 1 and 2 wt% filler in the biocomposites. The physico – mechanical, thermal characteristics and magnetic properties of fabricated (OPAC)/Fe₃O₄ filled biocomposites have been measured by using DSC, TGA, DMA and VSM. A slight improvement in thermal stability was noticed for (OPAC)/Fe₃O₄ loaded biocomposites. The PU/(OPAC)/Fe₃O₄biocomposites are thermally stable up to 250°C and completely degraded above 520 °C. The biocomposites degradation kinetic parameters for each step of the thermal degradation processes have been studied using two mathematical models namely, Coats–Redfern and Broido's methods. The PU/(OPAC)Fe₃O₄ biocomposites showed high mechanical, magnetic, thermal properties at a low loading of 2 wt.%, and could potentially be used for a wide range of applications.

Keywords

PU/(OPAC)/Fe₃O₄; biocomposite; orange peel activated carbon; kinetic parameters

1.INTRODUCTION

The polymer green composites are one of the important material due to eco-friendly, cheaper and partly replacing mineral fillers. However, with increasing use lingo cellulosic sources in the composite industries, overall manufacturing costs decrease. In the recent years, many research reported that agricultural wastes material such as bagasse sugarcane, coconut shell powder, sawdust and Pomegranate peel powder were used as waste material for improving physical and thermal properties of matrix polymer [1, 2].The nanocomposites including nanoparticles do not have excellent physical properties, like elongation at break, tensile modulus, solvent resistance, and tensile strength[3], but possess functionalities, involving biomaterials[4].

Nano metal oxides with different morphology such as nanocrystals, nanorods, nanowires, nanotubes and nano flowers have been reported and all these types have not sustained its own properties after they are covered/coated by external materials like activated carbon and carbon nanotube[5].

Hence, today, new material as like (OPAC)/Fe₃O₄ biomaterial is used for expand biocomposites in the composite industries. In recent years, some researches have been reported that the carbon-coated Fe₃O₄ composites with waste agricultural material have good magnetic behavior, chemical

and electrical properties [6]. Recently, multiple polymers were fabricated as magnetic polymer nanocomposites, which polyurethane is preferred due to has excellent mechanical properties, good handling, chemical stability, and very low-cost products[7].

The main goal of present work is the obtainment and characterizations of PU/(OPAC)Fe₃O₄ biocomposites, to the best of our knowledge, (OPAC)/Fe₃O₄ filled PU biocomposites have not been reported yet. The new magnetic biocomposites materials were obtained by physical mixture of polymer and filler and characterized regarding the biocomposites. As most of nutraceutical industries are localized, these nutraceutical residues may contribute to environmental pollution. Hence in this research article an attempt has been made to utilize this agro-industrial waste and nontoxic environmentally friendly to prepare green composites which has little or no commercial value.

2.EXPERIMENTAL

2.1.Materials

Ferric chloride hexahydrate (FeCl₃ · 6H₂O), FeCl₂ · 4H₂O, Tetrahydrofuran (THF) , and NaOH were purchased from Jahan e shimi Orumiya Chemical Reagent Co., Ltd. Toluene diisocyanate (TDI) was purchased was obtained from Sigma, Jahan e shimi

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Orumiya (Iran). The Jagropol oil (115) was obtained from Jahan e shimi Orumiya. Dibutyltindilaurate (DBTL) ($C_{32}H_{64}O_4Sn$), was purchased sigma Alderich.

2.2. Synthesis of orange peel activated carbon (OPAC)

For preparation (OPAC), at first orange peel was collected, washed and was dried at room temperature. Then it put at $110\text{ }^\circ\text{C}$ for 12 h in a Hot Air Oven, after made powder by a mixer and sieved using a 30 mm mesh size ($50\text{ }\mu\text{m}$). It was carbonized at $450\text{ }^\circ\text{C}$ during 2h under a nitrogen atmosphere. It was mixed with activating agent potassium hydroxide (KOH) (6 M) in the during 3h at room temperature, and dried at $120\text{ }^\circ\text{C}$ for one day. The mixture set a tubular furnace at $500\text{ }^\circ\text{C}$ for 4 h. In the end, activated carbon washed with concentrated hydrochloric acid and deionized water.

2.3. Synthesis of magnetic orange peel activated carbon (OPAC)/ Fe_3O_4

The 1 g of the orange peel activated carbon (OPAC) was dispersed in 40 ml ethanol containing 3g of $FeCl_3 \cdot 6H_2O$ and 6g of $FeCl_2 \cdot 4H_2O$ with molar proportion of 1:2 were dissolved in deionized water at room temperature. NaOH solution (3 mol·L⁻¹) is added and stirred for 30 minutes, and sonicated for 10 minutes. 10 mL of $NH_3 \cdot H_2O$ 2M is added during the reaction to obtain black precipitate and washed with distilled water several times. The sample was obtained as magnetic orange peel activated carbon (OPAC) Fe_3O_4 [8].

2.4. Formation of PU/(OPAC)/ Fe_3O_4 biocomposites

The PU/(OPAC)/ Fe_3O_4 biocomposites were synthesized by the intercalation of PU onto orange peel activated carbon (OPAC)/ Fe_3O_4 via in-situ polymerization manner [9]. Jagropol oil (0.001mol) was initially dissolved in 100 ml of tetra hydro furan (THF) including with different weight fractions of orange peel activated carbon (OPAC)/ Fe_3O_4 viz., 0, 0.5, 1 and 2 wt.% and placed in three-necked round bottomed flask. The content was stirred for 30 min or until the uniform mixture was obtained. This salvation was followed by adding the toluene diisocyanate (TDI) (0.002 mol) and 2 to 3 drops of DBTL as catalyst. The reaction mixture of the flask was stirred continuously for about 1 h under oxygen free nitrogen gas purge at $60\text{--}70\text{ }^\circ\text{C}$. The reaction mixture was poured into a cleaned and releasing agent the mould was kept in preheated circulating hot air oven at $70\text{ }^\circ\text{C}$ for 8–10 h. The toughened PU biocomposite sheet thus formed was cooled slowly and removed from the mould. The above procedure was repeated for different weight percent of (OPAC)/ Fe_3O_4 viz., 0,

0.5, 1 and 2 wt.%. The synthesis procedure is presented in Fig 1. coated glass mould and allowed to stand for 12 h at room temperature.

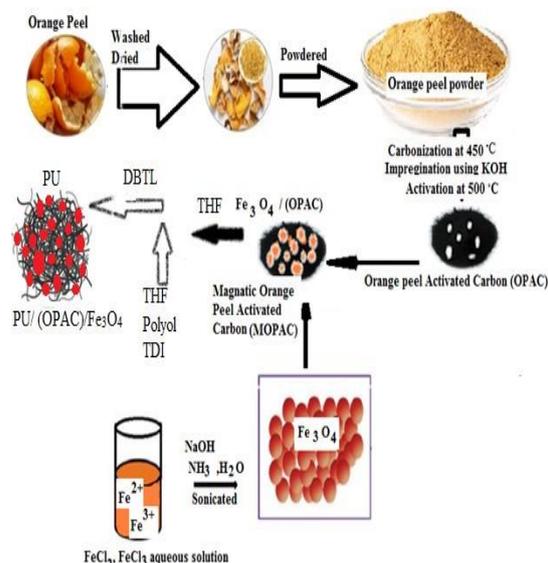


Fig. 1. The schematic for the formation of PU/(OPAC)/ Fe_3O_4

2.5. Treatment of TGA data

The thermal properties such as percentage weight loss and thermal degradation parameter of PU/(OPAC)/ Fe_3O_4 biocomposites were analyzed by using DuPont TA Instrument with TGA-Q 50 module at a heating rate of $20\text{ }^\circ\text{C}/\text{min}$ in N_2 atmosphere. The kinetic data of bare polyurethane and its biocomposites were calculated by using Coats–Redfern [10] and Broido's [11] methods. Coats–Redfern (CR) [24] is respected as follows;

$$\log(-\log(1-\alpha)/T^2) = (\log(AR/\beta Ea)) - (Ea/2.303RT) \quad (1)$$

where, T , α , β , A , R and Ea are the derivative peak temperature, the fraction of sample decomposed at time T , the heating rate, the frequency factor, gas constant and the activation energy, respectively.

The slope of activation energy is obtained by plotting of $\log\{-\log(1-\alpha)/T^2\}$ versus $1/T$.

Broido's (BR) [11] method express as follows;

$$\log(-\log(1-\alpha)) = -(Ea/2.303R)((1/T) + K) \quad (2)$$

where, T , $(1-\alpha)$, Ea and R are the peak temperature of derivative curve of TG, the fraction of number of initial molecules not yet decomposed, the activation energy and the gas constant, respectively. The surface functional groups of Fe_3O_4 nanoparticles and PU/(OPAC)/ Fe_3O_4 biocomposites have been discovered by applying FTIR spectrometer. The magnetic of samples were obtained by using vibrating sample magnetometry (VSM) at room temperature in a maximum magnetic field of 16 kOe.

Table 1. Physico-mechanical properties of biocomposites

Properties		(OPAC)/Fe ₃ O ₄ content (wt%)			
		0	0.5	1	2
Density (g/cc)	Exptl.	1.0708	1.0937	1.1452	1.2875
	Theo.	-	1.1050	1.1640	1.3500
Surface hardness (shore A)		70	75	82	87
Tensile strength (σ) (MPa)		5.0	6.58	7.44	7.14
% Improvement in tensile strength		-	24	32	30
Tensile modulus (MPa)		0.18	0.29	0.34	1.15
Elongation at fracture (e) (%)		77	140	145	112
Product (σe)		385	921	1078	799

3. RESULTS AND DISCUSSION

3.1. Physico-mechanical properties

The physico- mechanical properties for matrix PU and PU biocomposites have been cleared in Table 1. The density of (OPAC)/Fe₃O₄ and PU/(OPAC)/Fe₃O₄ was a range 1.0708 and 1.2875 g/cc, respectively. The measured density values for all PU/(OPAC)/Fe₃O₄ biocomposites are given in Table 1. The table 1 is shown which, the density of the (OPAC)/Fe₃O₄ filled PU biocomposites enhancement as increment in (OPAC)/Fe₃O₄ value that, is due to enhance in great dense filler in light dense bare PU. The experimental values in the contrast theoretical values less and also with increasing (OPAC)/Fe₃O₄ values, it enhanced. This can be attributed to that there is a poor interaction between PU matrix and (OPAC)/Fe₃O₄ filler.

3.2. Surface hardness

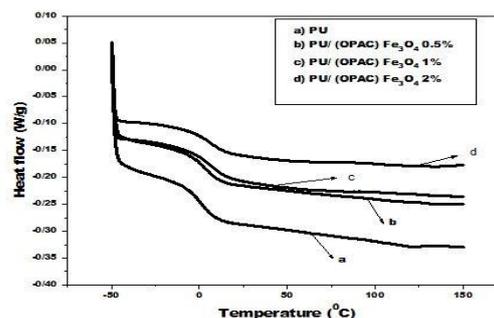
From table 1 is noticed that the surface hardness values for experimental bare PU and its biocomposites line in the range 70-87 shore A. The shore A of biocomposites increase with increasing (OPAC)/Fe₃O₄ values. Also, this is due to rigid (OPAC)/Fe₃O₄ content in the biocomposites as expected. This consequence showed that biocomposite have good dimensional stability than the bare polymer.

3.3. Tensile behavior

The table 1 is shown that tensile strength of PU/(OPAC)/Fe₃O₄ higher than the tensile strength of bare PU. Also, it indicated that after increase 2wt% of (OPAC)/Fe₃O₄ into biocomposites, it remains constant. This is due to interaction between nanoparticles and filler is going be aggregation and as result it remains reduce or constant in tensile strength. The percentage elongation at fracture of matrix PU and PU/(OPAC)/Fe₃O₄ lie in the range 77 to 145 that it increase 47 % and 32 % for biocomposite involving 1 % and 2 % of (OPAC)/Fe₃O₄. This results obtained due to action filler and matrix polymer and decrease network mobility because of higher filler loading. In ordinary, the modulus of biocomposites increment with increase stiffer filler [12]. From table 1 obtained that the tensile

modulus of biocomposites and bare PU lies in the range 0.18 – 1.15 MPa. This results depend hollow particles, cavity and on the particle wall thickness [13, 14]. At the end, the data of physico- tensile properties indicated that 1% (OPAC)/Fe₃O₄ filled PU biocomposites referred as the optimized composition. Comparing the results it can be seen that 1% (OPAC)/Fe₃O₄ filled PU biocomposite exhibited the higher tensile strength, elongation at fracture and tensile modulus values as compared to other formulations. Hence, 1% of (OPAC)/Fe₃O₄ loaded biocomposite is referred as the optimized composition.

This is due to the failure along the loading direction, the dispersed (OPAC)/Fe₃O₄ and cracks along the loading direction [15]. Lancaster [16] stated that the product of σe factor (where, σ is the ultimate tensile strength and e is the elongation at fracture) is a very important factor which controls the abrasive behavior of composites. From the table it can be seen that the σe factor increases with increase in (OPAC)/Fe₃O₄ content up to 1 wt % and it lies in the range 385 - 1078. Higher the values of σe , higher will be the wear resistance of the composites.

**Fig. 2.** DSC thermograms of biocomposites

3.4. Differential scanning calorimetry

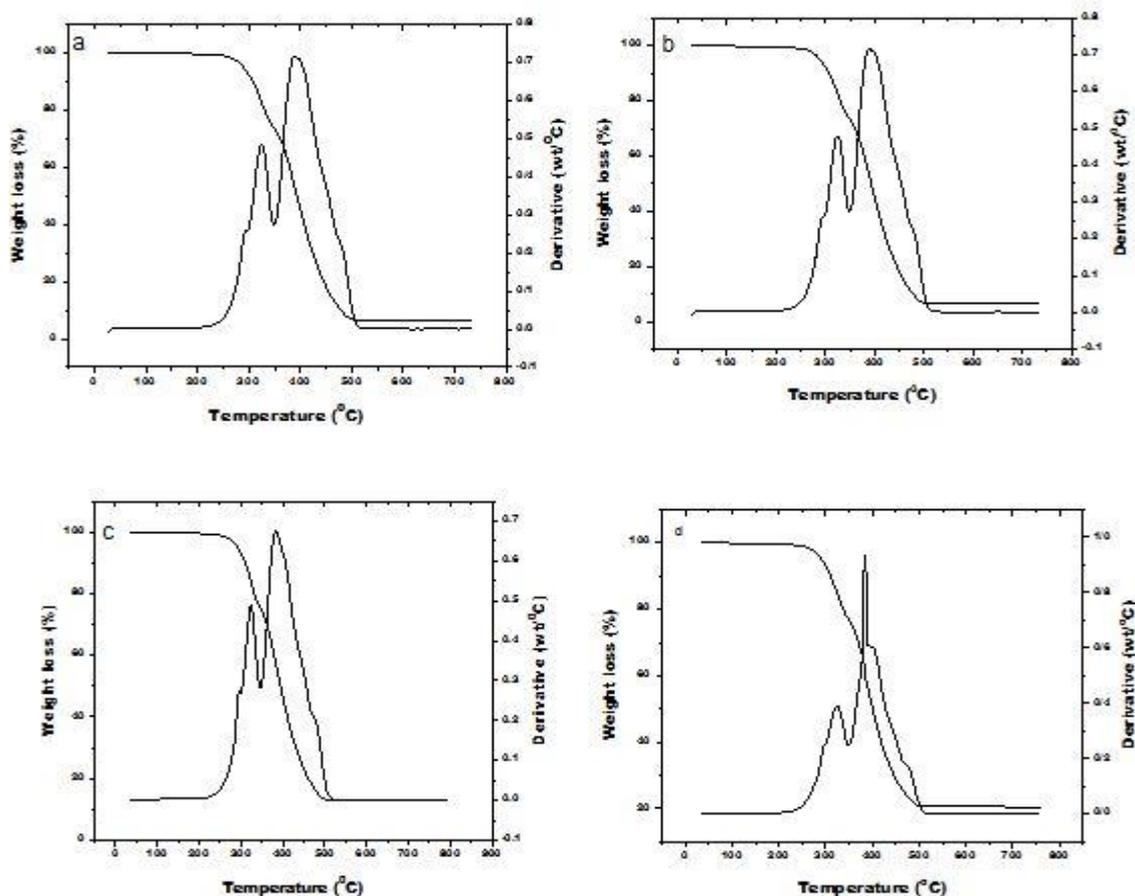
For analysis glass transition temperature (T_g) of bare polyurethand and PU/ (OPAC)/Fe₃O₄ biocomposites, ara using Differential scanning calorimetry (DSC), that DSC thermograms those are offered in Figure 2 and tabulated in Table 2. From the thermograms it can be seen that the T_g values near same and do not have significant change.

Table 2. Tg of PU and PU/(OPAC) Fe₃O₄ biocomposites

(OPAC)/Fe ₃ O ₄ content (wt.%)	Tg(OC)
0	-6.5
0.5	-6.1
1	-5.6
2	-5.7

3.5. Thermogravimetric analysis

For study the effect of (OPAC)/Fe₃O₄ on the comparative thermal stability of biocomposites, TGA thermograms along with derivative curves and TGA measurement of bare PU and their biocomposites have been given in Figure 3 (a)-(d) and Table 3. It was perceived which the thermal degradation template was approximately same for all (OPAC)/Fe₃O₄ filled its biocomposites and it was noticed that all the samples follow two stage thermal degradation processes.

**Fig. 3.** TGA thermograms of; (a) PU, PU with; (b) 0.5 %, (c) 1 % and (d) 2wt % (OPAC)/Fe₃O₄ content**Table 3.** Thermal data obtained from TGA thermograms for PU and biocomposites

(OPAC)/Fe ₃ O ₄ content (wt %)	Degradation stages	Temperature (OC)+ 2			Weight loss (%)
		Ti	Tmax	Tf	
0	1	255	318	345	27.6
	2	345	380	511	71.2
	Ash	-	-	-	1.2
0.5	1	254	322	350	26.8
	2	350	399	517	67.2
	Ash	-	-	-	6.0
1	1	254	322	342	24.4
	2	345	384	515	62.8
	Ash	-	-	-	12.8
2	1	250	321	348	22.9
	2	348	383	510	56.5
	Ash	-	-	-	20.6

As, it can be seen that weight loss for the first step thermal degradation process of bare PU and 2% (OPAC)/Fe₃O₄ filled PU biocomposite were 27.6% and 19.4% respectively. It occurred due to the soft segment of PU and volatile impurities. The chief pyrolysis produce could be carbon dioxide [15]. In the second stage, the chief weight loss for bare PU and 2% of (OPAC)/Fe₃O₄ filled PU composite was 71.2 % and 48.6 %, in the temperature range 350-5160C. It may be because of nitriles of aromatics, carbon dioxide, Fe-O, liberation of HCN and de cross linking of PU composites[9].The weight loss in both degradation steps decreases with increase in (OPAC)/Fe₃O₄value. From the figure it can be seen that the weight patently loss with increment in (OPAC)/Fe₃O₄ value. The onset of decomposition of whole samples were nearly similar (250-255 0C).

3.6. Kinetic analysis of thermal degradation

The plots of ln[-ln(1-α)] versus 1/T (BR) and ln[-ln(1-α)]/T² versus 1/T (CR) for first and second

stage degradation procedure of (OPAC)/Fe₃O₄ filled PU composites are presented in Figures 4 (a-b) and 5 (a-b) respectively. The R2 values and calculated activation energy (Ea) for every thermal degradation procedure and for every system are appointed in Table 4.

The results obtained of figure show that the activation energy after incorporation of (OPAC)/Fe₃O₄ filler lies in the range 133–175 and 124–193 kJ/mol for Broido and Coats–Redfern methods and reduce for first step degradation process. In spite of, the activation energy no change systematic variation for all samples. Also, the Ea values for the first step thermal degradation process is lower than in secondary step degradation for both the methods for all samples because it needs less energy for removing volatile components and low molecular weight materials (soft component). But Ea for second step degradation process need higher energies due to is required high energy because of bond scission and unzipping of PU chains.

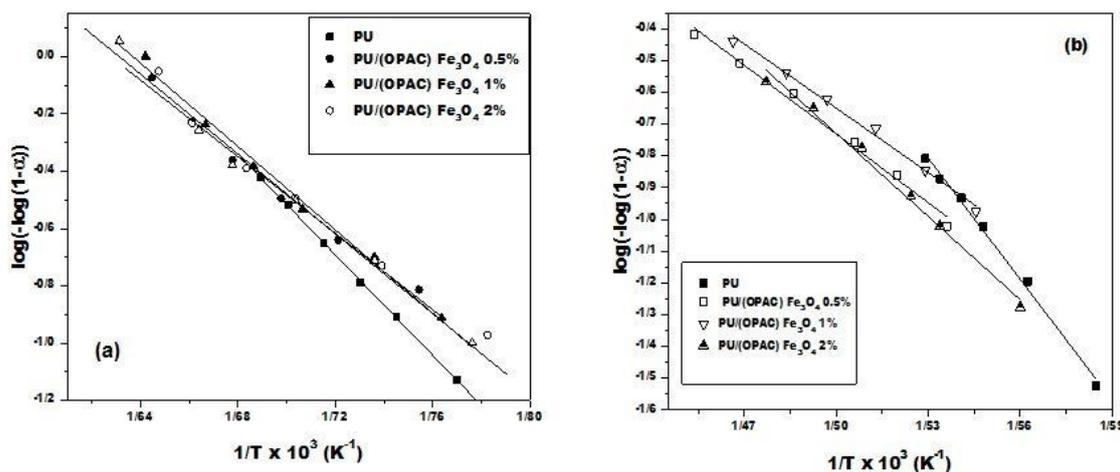


Fig. 4. Broido plots for; (a) first step and (b) second step thermal degradation process of biocomposites

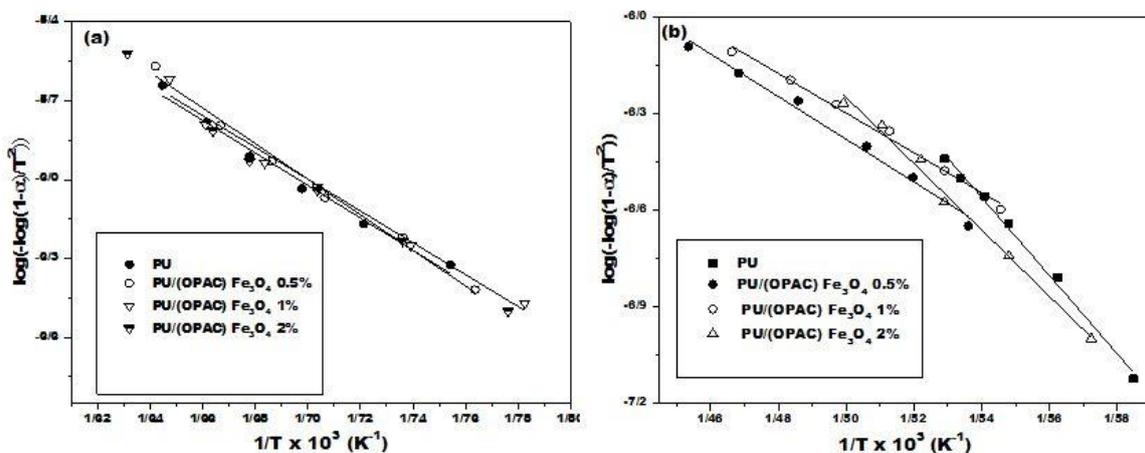
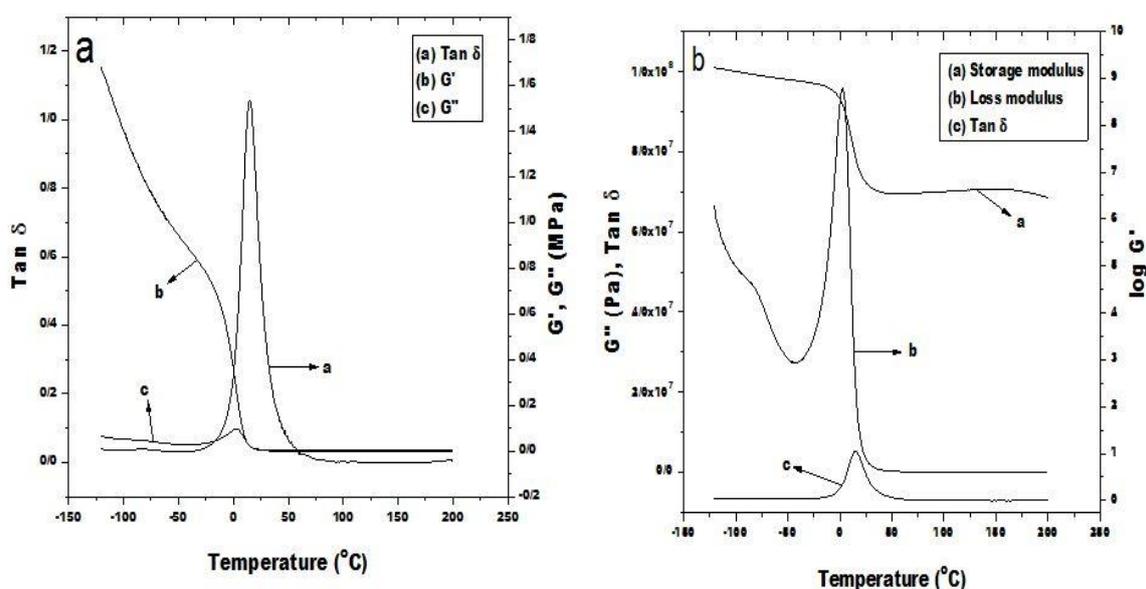


Fig. 5. Plots of Coats-Redfern method for (a) first step and (b) second step thermal degradation process of biocomposites

Table 4. Activation energies calculated by CR and BR methods with respective concurrency value (R2) for PU biocomposites

(OPAC)/Fe ₃ O ₄ content (wt %)	Degradation stage	Activation energy (Ea) (kJ/mol)			
		CR	R2	BR	R2
0	I	193	0.9996	175	0.9989
	II	240	0.9958	251	0.9952
0.5	I	126	0.9921	134	0.9979
	II	135	0.9895	185	0.9986
1	I	136	0.9965	147	0.9945
	II	125	0.9954	136	0.9955
2	I	124	0.9898	133	0.9923
	II	205	0.9939	215	0.9929

**Fig. 6.** (a). DMA curves of 0.5wt % (OPAC)/Fe₃O₄filled PU biocomposite, (b) Log G', G'' and Tan δ curves of 0.5 wt % (OPAC)/Fe₃O₄ filled PU biocomposite**Table 5.** Data obtained from DMA thermograms for PU biocomposites

(OPAC)/Fe ₃ O ₄ content (wt %)	Tan Delta (Peak Max)		T _g (0C)
	Experimental	Theoretical	
0	1.04	1.04	14
0.5	1.05	1.02	13
1	1.03	0.94	11
2	0.91	0.86	16

3.7. Dynamic mechanical analysis

Dynamic mechanical measurements applied for consider a variation in dynamic storage modulus, loss modulus and lateral groups damping ($\tan \delta$) expresses an ability of converting the mechanical energy into heat energy. It is defined as;

$$\tan \delta = G'' / G' \quad (3)$$

where, $\tan \delta$ is phase angle between stress, G'' is elastic loss modulus and G' is the elastic storage modulus. As a consequence, $\tan \delta$ is a main parameter describing material's viscoelastic behavior[17]. Figure 6 (a)-(b) exhibit the temperature dependence of G' , G'' and $\tan \delta$ for 0.5 wt % (OPAC)/Fe₃O₄ filled PU composite the temperature range from -50 to 40 0C, that the

elastic storage modulus (G') with increase temperature reduce . The $\tan \delta$ was shown that values gain in the initial stage and then decline with increasing temperature. With adding up to 1 wt % (OPAC)/Fe₃O₄ into matrix polymer, the $\tan \delta$ values of the biocomposites remains constant (Table 5) that it shows which the damping property of the PU biocomposites retains.

However, with increasing volume fraction (OPAC)/Fe₃O₄, the matrix viscoelasticity and $\tan \delta$ values accordingly reduce[18]. The Tan delta measurement in table 5 show that there is a small change in the T_g, and no systematic variation in T_g with increase in (OPAC)/Fe₃O₄ content. Also,

the tan delta values measured theoretically is some less than of experimental values.

3.8. Magnetic properties of PU/(OPAC) Fe₃O₄ biocomposites

The magnetic properties of PU/(OPAC)Fe₃O₄ biocomposites have been analyzed by using a vibrating sample magnetometer (VSM). From figure 7, it can be seen that magnetic properties biocomposites improved by increasing of (OPAC)Fe₃O₄ content. The superb magnetism was chiefly due to the good dispersion of (OPAC)Fe₃O₄. This reduction in saturation magnetization was due to the introduction of a non-magnetic PU-coated shell. The magnetic properties of biocomposites are effected by surface disorder, distribution, and grain size that it caused in an betterment of in the magnetism of the biocomposites[19].

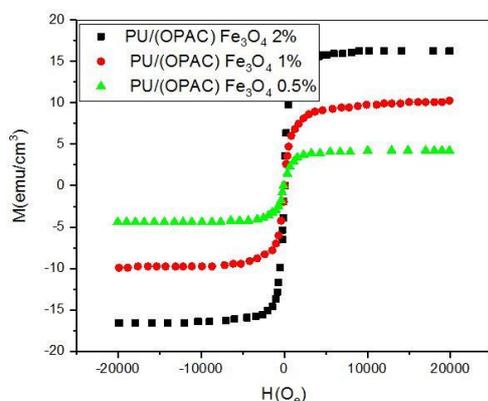


Fig. 7. The hysteresis loops of PU/(OPAC)/Fe₃O₄ films with different (OPAC)/Fe₃O₄ contents.

5. CONCLUSION

PU composites with different weight fractions of (OPAC)/Fe₃O₄ have been prepared and characterized for mechanical and thermal behaviors. The density of PU composites increased with increase in (OPAC)/Fe₃O₄ content. The tensile strength, percentage elongation at break and σ_e factor was maximum for 1 % (OPAC)/Fe₃O₄ loaded biocomposite. Tensile strength increased from 5.0 MPa for unfilled PU to 7.2 MPa for 1 % (OPAC)/Fe₃O₄ filled PU biocomposite and later it starts declining with increase in (OPAC)/Fe₃O₄ content. The dimensional stability and tensile modulus improved prominently with increase in filler content from 0 to 2wt%. DSC and DMA studies indicate that the influence of filler on T_g was insignificant. TGA data reveals that, improvement in thermal stability of PU as increase in (OPAC)/Fe₃O₄ loading. Activation energy of thermal degradation process was calculated using two mathematical modeling. The activation energy for the first step was found to be lower than the second step. The saturation magnetization

increased as the magnetic (OPAC)Fe₃O₄ loading increased and reached up to 14.22 (emu/cm³) when the (OPAC)Fe₃O₄ content was 2%.

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سینتیک تخریب حرارتی پلی اورتان / کربن فعال مغناطیسی مشتق شده از پوست پرتقال بیوکامپوزیت ها $(OPAC)/Fe_3O_4$

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چکیده

در کار حاضر، کربن فعال مشتق شده از پوست پرتقال پر شده از $(OPAC) / Fe_3O_4$ در بیوکامپوزیت های پلی اورتان با درصد کسرهای وزنی مختلف از $(OPAC)/Fe_3O_4$ ، ۰.۵، ۱ و ۲ درصد وزنی در بیوکامپوزیت ها سنتز شد. ویژگی های خواص مکانیکی، حرارتی و مغناطیسی بیوکامپوزیت های پر شده از $(OPAC)/Fe_3O_4$ با استفاده از DSC، TGA، DMA و VSM اندازه گیری شده اند. بهبود جزئی در پایداری حرارتی برای بیوکامپوزیت های بارگذاری شده از $(OPAC)/Fe_3O_4$ مشاهده شد. کامپوزیت های زیست پلیمری PU/ $(OPAC)/Fe_3O_4$ تا دمای ۲۵۰ درجه سانتیگراد از نظر حرارتی پایدار هستند و دردمای بالاتر از ۵۲۰ درجه سانتیگراد کاملاً تخریب می شوند. پارامترهای جنبشی برای تخریب بیوکامپوزیت ها برای هر مرحله از فرآیندهای تخریب حرارتی با استفاده از دو مدل ریاضی یعنی روش های Coats-Redfern و Broido مورد مطالعه قرار گرفت. بیوکامپوزیت های پلی اورتان پر شده با ۲ درصد وزنی از $(OPAC)/Fe_3O_4$ ، خواص مکانیکی، مغناطیسی و حرارتی بالایی را در برابر با درصد وزنی کم نشان دادند. که این می تواند به طور بالقوه برای طیف وسیعی از کاربردها مورد استفاده قرار گیرند.

کلید واژه ها

بیوکامپوزیت؛ PU/ $(OPAC)/Fe_3O_4$ ؛ کربن فعال مشتق شده از پوست پرتقال؛ پارامترهای سینتیکی