



Mechanical and Thermal Degradation Kinetic Study Of Basalt Filled Polyvinyl Chloride Composite Material



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Abstract

Polymeric composites filled with natural fillers are an emerging area in polymer science, and have recently attracted the attention of researchers. Because of their relatively good service properties, the major contributions of these natural fillers are lowering the cost of composite materials and producing faster molding cycles. In this study, the influence of micro particulate ground basalt rock addition on physical, mechanical, and thermal kinetic properties of polyvinyl chloride (PVC) reinforced basalt rock powder (BRP) composite material was investigated.

Results showed that basalt rock powder (BRP) affect the physical, mechanical and thermal properties of the (PVC/BRP) composites. Increasing the BRP loading will produce a relative drop in water absorbability, also decrease the tensile strength and elongation at break, while causing an increase in hardness shore A and compression strength values. Moreover, thermal stability, activation energy, and entropy were increased with increasing BRP as proven by Thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC).

Keyword: Polyvinylchloride composite; Basalt rock; Thermal degradation; Mechanical properties; Thermal stability; Activation energy; Entropy.

Introduction

Polymers are very vital in our life. For so many years, people used polymers for manufacturing goods and articles needed for a civilized life. All material scientists, chemists, physicists, technologists, chemical and mechanical engineers, pharmacists and specialists in other disciplines are seriously participating in the development programs of polymer science [1]. Thus, polymer and polymer chemistry play a significant role in the development of new areas in fields of pharmacy, biomedicine, molecular biology, biochemistry, biophysics and industry [2, 3]. This is made possible due to the fact that ease of processing, productivity, and cost reduction all are interesting advantages of using polymers. In most of these applications, the

characteristics and properties of polymers are modified and tailored using fillers and fibers to meet the demands of high strength and high modulus [4, 5].

PVC is one of the most important polymers, which is used in many fields. However, PVC and its composites are subject to certain limitations in many applications because of its inherent drawbacks, such as poor thermal stability and brittleness. [6]. Another interesting point is that, PVC can be easily mixed with other polymers, to form polymer alloys, due to its high polarity and compatibility with a wide range of other high-performance polymeric materials. The poor properties of PVC can be improved, with the addition of additives and copolymerization with other monomers [7]. PVC constitutes the most used thermoplastic material as regards the worldwide polymer consumption. It is used in a variety of applications, such as , medical, industrial

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Receive Date: 12 July 2020, Accept Date: 27 September 2020

DOI: 10.21608/EJCHEM.2020.35343.2739

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,construction, domestic materials , electrical insulation , pipes for diverse applications , packaging and food industry , surface protecting films etc. [8-11]. To improve the processability of PVC and to prevent its degradation, different additives must be used with its formulation. The additive types and the PVC formulations depend manly on the final application of the fabricated product, and the employed processing technique. These additives are; external, internal and heat stabilizers, plasticizers, and fillers for modifying pvc properties and lowering the cost of its composites [12].

Basalt rock (BR) is an igneous rock with about 45-53% silica (SiO_2) and the feldspathoid constitutes less than 10% by volume. While at least 65% of the rock content is feldspar [13]. This volcanic rock has high strength and density, as well as high chemical properties, resistance to flames, mechanical strength, durability, and the capability to insulate heat and sound. Using inorganic fillers to modify thermoplastic and thermoset polymers can lead to tailored specifications which suitable numerous applications of polymeric composites. Although there several studies in this regard, but the horizons are still open to discover new advanced polymeric materials improved performance characteristics. Basalt composites are applied in diverse ways: to reinforce plastics and polymers; strengthen soil, bridges, and roads; manufacture industrial floors; isolate heat and sound in residential and industrial buildings [14]. The incorporation of basalt rock powder (BRP) as inorganic filler in the polymeric composite materials is of current interest. Despite so many works on the application of BRP as low-cost filler with polymers such as poly ethylene, poly propylene, epoxy and unsaturated polyester to formulate composites with excellent properties [15-17], but to the best of the authors' knowledge, no study has been conducted on applying basalt rock powder as inorganic filler for PVC. This work aimed to study the influence of adding varying ratios of basalt rock powder (BRP) on physical, mechanical and thermo kinetic properties of PVC reinforced BRP composites.

Experimental part

Materials:

A white suspension powder general-purpose polyvinyl chloride (PVC) resin, with k value of 70 was used as a base resin, supplied by sabic company Saudi Basic Industries Corporation. A plasticizer, diethylhexyl phthalate (DEHP) from Hubei ocean biotech co. ltd, china. Lead stabilizer dibasic lead sulphate (DBLS), internal lubricant calcium octadecanoate (COD) and heat stabilizer

octadecanoic acid (ODA) from Merck. Basalt rock, supplied by the ministry of industry and minerals – Iraq.

Experimental method

Preparation of BRP:

Basalt rock was washed, dried, and then grinded to fine powder at a particle size of (0.29-14.58) μm as shown in (Fig.1), by using a ring mill machine. The chemical composition of basalt rock powder was analyzed in Directorate of materials research, science and technology, Baghdad, Iraq as illustrated in table 1.

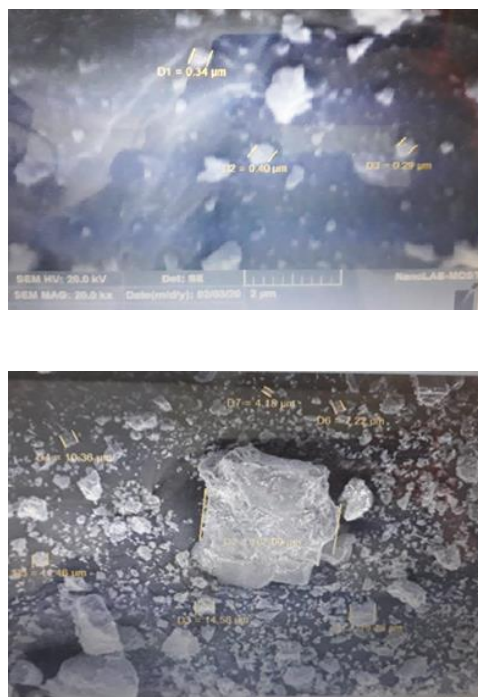


Fig. 1. Grinded basalt rock

Table 1

Chemical composition of BRP

Composition	Wt%
SiO_2	41.53
Al_2O_3	18.56
Fe_2O_3	13.87
CaO	12.55
MgO	2.73
Na ₂ O	3.60
K ₂ O	1.37
($\text{SiO}_2 + \text{Al}_2\text{O}_3$)	0.65

Compounding of PVC

A high-speed laboratory mixer (Brabender micron 50 gm) was used in the compounding process. All the powder additives DBLS, COD and ODA were mixed for 5 minutes at 50°C and 10 rpm. Then the premixed powders and the DEHP were added to the PVC and filler in the mixing chamber and mixed at 95°C and 45 rpm for 10 minutes. The produced dough was then molded by compressor at a 10 bar pressure for 10 minutes. The resulting polymeric composite sheet then machined to the desired final dimension according to the required test. Table 2 shows the formulation of varying ratios of BRP used in the preparation of PVC based composites. Three specimens of each formulation were taken and were analyzed for determining the mechanical, physical and thermal properties.

Mechanical measurements

The tensile measurements and elongation at break were carried out according to ASTM D638, using Universal Testing Machine JIANQIAO testing equipment CZL203-200Kg, China. Shore (A) test was performed by using a Durometer device, Shore hardness tester, time group Inc. TH200, iso 9001, Italy. Compressive strength was carried out with Carver-D695 Farrell Instruments - LTC, compression machine. Each sample was pressed at room temperature and a compression force (0 – 1700N) for 5 minutes. The compression pressure (1700N) was the maximum pressure reached for the test sample.

Thermal measurements

Thermal conductivity of all specimens was measured by using Lee's Disc device (Griffin & George/England). Three copper discs (A, B, C) of 40 mm diameter x 12.25 mm thickness each were used. The composite samples were machined to diameter 40 mm and 3mm thickness. The test was carried out according to ASTM D 150. Thermal decomposition of polymeric composite samples helps to study the thermodynamic, kinetic parameters, optimum limit temperature of the process, and thermal stability. The thermo gravimetric measurements were carried out according to ASTM E-1131 by a Perkin Elmer TGA analyzer, under nitrogen gas at 10 Co/min rate of heating. DSC test was carried according to ASTM D-3418-12.

Results and Discussion

The addition of basalt rock powder (BRP) at different ratios to polyvinyl chloride (PVC) is cost effective and can improve its thermomechanical performance. In this study, a composite material

consists of basalt rock reinforced polyvinyl chloride is developed. Table 2 shows the different formulations of PVC filled basalt composite material

Mechanical properties

In this study different mechanical properties like tensile strength, elongation at break %, hardness (shore A), and compressive strength were performed in order to evaluate the mechanical characteristics of (PVC) composite materials filled with different percentage of basalt rock powder. The result yielded that inclusion of (BRP) gives an apparent influence on the composite mechanical properties. Tensile strength gives us information about the behavior of the composite material when it is subjected to pulling or stretching force before it fails. While elongation at break, means the capability of the tested specimen to overcome shape changes without cracking. Elongation is the ratio between increased and initial length after breakage of the specimen at a controlled temperature. The relationship between tensile and elongation is shown in the following equations :

$$\text{Tensile Strength} = \frac{\text{max force(N)}}{W*d}$$

$$\text{Elongation} = \frac{\Delta L}{L_0} * 100\%$$

Where = width, d = display, ΔL = displacement

It is clear from the data shown in table 3, that as the ratio of filler increases, tensile strength and elongation values decreases. This can be attributed to the restriction of polymer chain movements, because at higher BPR loading the PVC chain displacement and moment due to the applied force are reduced. At high filler concentration the restricted chain movement will produce stresses which in turn may generate interface cracking [18, 19]. The compression strength is the capability of the composite material to withstand the load tending to resize it. Based on the data shown in table 1, and due to the fact that the filler particles occupy the interstitials between PVC particles, the compression strength of polyvinyl chloride composite material is increased with increasing BRP filler concentration. This leads to decrease in void content, which increases by resisting failure and reshaping of the specimen, and hence an increasing in compression strength. The hardness shore A is a characteristic of the composite material expressing its resistance to indentation and penetration. It is performed according to ASTM D2240 at room temperature. From the data shown in table 3, also the hardness of the PVC composite material specimens are increased with increasing BRP concentration [20].

Table 2

PVC formulation using basalt rock powder (BRP) as filler

Sample No.	Concentration (%)					
	BRP %	PVC	DEHP	DBLS	ODA	COD
1	58.8	30	10	0.4	0.4	0.4
2	48.8	40	10	0.4	0.4	0.4
3	38.8	50	10	0.4	0.4	0.4
4	28.8	60	10	0.4	0.4	0.4
5	18.8	70	10	0.4	0.4	0.4
6	0	88.8	10	0.4	0.4	0.4

Table 3

Mechanical properties of PVC filled BRP composite material

BRP %	Tensile (MPa)	Elongation at break (%)	Compressive Strength (MPa)	Hardness (Shore A)
58.8	1.88	46.7	8.95	104.2
48.8	2.27	72.43	7.60	103.3
38.8	2.40	101.5	6.68	99.5
28.8	3.09	118.3	6.04	98.6
18.8	3.34	143.5	5.20	94.9
0	3.91	172.4	4.89	80.67

Table 4

Water adsorption for PVC filled BRP composite material

Sample NO.	Water absorption %		
	5 days	10 days	15 days
1	0.8	1.6	2.0
2	0.92	1.74	2.14
3	0.99	1.97	2.92
4	1.41	2.0	3.08
5	1.5	2.3	3.2

Water absorption

Water absorption of the specimens was carried out according to ASTM D570. Three test specimens of each formulation were dried in an oven for 24 h at $80 \pm 2^\circ\text{C}$. Then were weighed with a precision of 0.001 g and immersed in distilled water at ambient temperature for (5, 10, and 15 days). The specimens were removed from water, and dried with a cloth (lint free) and the wet weight values were determined. Water absorption percent was determined as follows:

$$W = \frac{W_{\text{final}} - W_{\text{initial}}}{W_{\text{initial}}} \%$$

Where

W_{initial} = initial weight before immersion

W_{final} = final weight after immersion.

Water absorption usually occurs at the composite materials outer layers and gradually decreases into the bulk of the matrix. High water absorption leads to a material with higher wet characteristics which causes, possible decrease in the composite strength, and increase in their deflection, swelling and other related characteristics. These can result in deterioration of mechanical properties of materials [21]. The water absorption percent of the examined specimens are shown in table 4. It is clear that the water absorption percent decreases with increasing BRP in the composite material. This is because the increasing filler particles will help to occupy the cavities and the free interspaces between PVC polymer chains which minimize the penetration of water particles through those chains. On the other hand the long immersion time will allow to more wetness and hence water absorbability of the

PVC- BRP samples increases with increasing immersion period.

Thermal properties

Thermal conductivity

The compounding process was performed in Banbury machine which facilitates the proper mixing of PVC with the additives and filler. The uniform distribution of the filler in the PVC matrix resulting in a stronger thermal network structure. Thermal conductivity is a key parameter in several thermal applications. It quantifies capability of the substances to conduct heat. It is very important to understand the characteristics of basalt in order to widen its applications [22]. The basalt rock thermal conductivity is about 1.4 (W/ m k) and this value depends on various parameters including mineral composition [23]. It is obvious from the data given in table 5 that the thermal conductivity increases with increasing filler concentration [24]. As the BRP fills the gaps or spaces between the PVC chains, they restrict the movement of the chains and improve their thermal properties. The results showed that the thermal conductivity shifted toward higher values as the BRP increased, this is in agreement with the literature [25].

Table 5

Thermal conductivity for PVC filled BRP composite material

BRP %	Thermal Conductivity K (w/m. k)
58.8	0.91839
48.8	0.76355
38.8	0.69377
28.8	0.64791
18.8	0.43765
0	0.28040

Thermal kinetic degradation

Fig 2. shows thermograph of PVC with a different weight percentage of Bazalt Rock Powder (BRP) of (58.8, 48.8, 38.8, 28.8, 18.8) weight percent, thermally decomposed under nitrogen atmosphere with a heating rate of 10 Co/min using TGA and DSC techniques. Thermal decomposition occurred in two steps, the first random scission and decyclisation occurs to polymer backbone chains followed by thermal volatilization of chars. However it can be seen that the curves were displaced to left - hand side at higher BRP contents because composites got higher thermal stability and higher BRP content retards the decomposition process. It can be seen the residue after decomposition of composite with content BRP of (58.8, 48.8, 38.8, 28.8, 18.8) weight percent were (59.99, 52.26, 28.88, 36.49, 27.96) weight percent respectively, however this indicates that mineral fillers act as a retarder on thermal

decomposition consequently thermal stability increased. This is in convenience with the previous literatures [26]. The typical DTG under nitrogen atmosphere at a heating rate of 10 Co/min are shown in Fig 3.

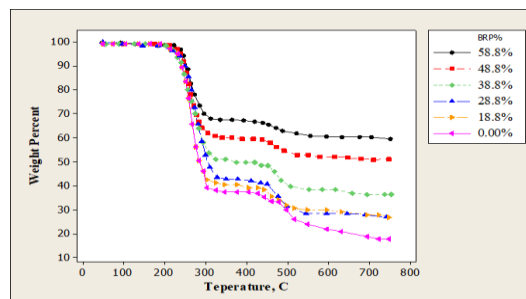


Fig.2. TGA profiles of thermal decomposition PVC/BRP

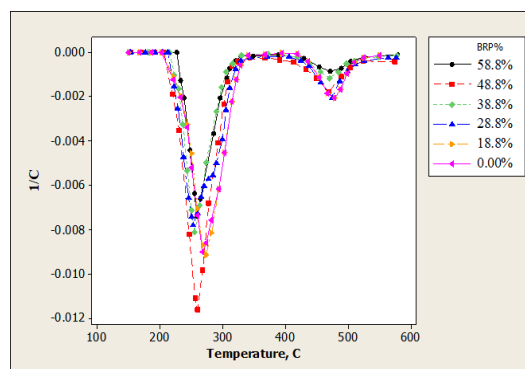


Fig.3. DTG profiles PVC/BRP composites in nitrogen flow

Table 6 shows weight percent losses during thermal decomposition of composites under different level of BRP content. At higher level of BRP decomposition occurs at a higher temperature compared with lower level content of BRP which showed weight losses occurs at certainly low temperatures, this is because of the increased thermal stability and the role of BRP particles in retarding of thermal decomposition. This condition occurs in all experimental work since polymer backbone chains become more rigid by filling the spaces, consequently intra Vander wall forces will arise between chains.

The kinetics of thermal decomposition can be obtained by using the following equation [27].

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \quad (1)$$

(α) represents a mass fraction loss during thermal decomposition and its value obtained from TGA chart, $k(T)$ is reaction rate constant and $f(\alpha)$ is reaction model of thermal decomposition of composites. $k(T)$ is Arrhenius equation:

$$\frac{d\alpha}{dt} = A \exp\left(-\frac{E}{RT}\right) f(\alpha) \quad (2)$$

$$g(\alpha) = A \exp\left(-\frac{E}{RT}\right) t \quad (3)$$

Equation 2 can be written as follow:

$$\frac{d\alpha}{dt} = \left(\frac{A}{\beta}\right) \exp\left(-\frac{E}{RT}\right) f(\alpha) \quad (4)$$

On integration of equation 4 and rearranging

$$g(\alpha) = \left(\frac{AE}{RT}\right) P(X) \quad (5)$$

Where $p(x)$ has many approximation with The Coats-Redfern (CR) method which is a non isothermal integral method using Mapel first order reaction model [27].

$$\frac{\ln g(x)}{T^2} = \frac{\ln AR}{\beta E} \left(1 - \frac{2RT}{E}\right) - \frac{E}{RT} \quad (6)$$

$$g(x) = (-\ln(1 - \alpha)) \quad (7)$$

Where

α = The extent of the reaction

β = The constant heating rate

A = The frequency factor

R = The universal gas constant

T = The decomposition temperature

E = The activation energy

By plotting $\frac{\ln g(x)}{T^2}$ against $\left(\frac{1}{T}\right)$ for each heating rate gives a family of straight lines of slope $\left(-\frac{E}{R}\right)$. Frequency factor directly determined from Y axis intercept by substituting values of activation energies (Fig. 4):

$$\text{Intercept} = \frac{\ln AR}{\beta E} \left(1 - \frac{2RT}{E}\right)$$

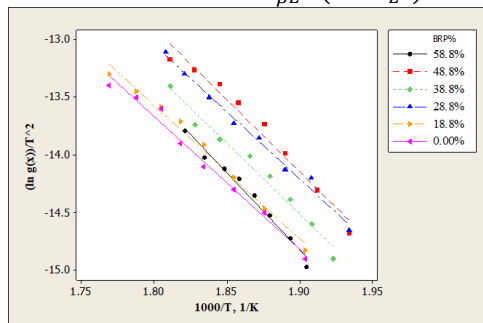


Fig.4. A plot of thermal decomposition of PVC/BRP composites utilizing coast-redfern method

Table 7 shows the thermal decomposition of PVC/BRP composites, and kinetic parameters at heating rate of 10 C°/min. It can be seen that the activation energy increases with increasing of BRP ratio, leading to an increase in the thermal stability of composites specimens. As mentioned before the decomposition occurs at two steps therefore peak temperatures were tabulated with reaction rate constant.

The thermodynamic properties were estimated by the following equations [28, 29].

Where ΔH is activation enthalpy, ΔS is activation entropy, ΔG is activation free energy of decomposition, T is maximum peak temperature, h is Plank constant and k is Boltzmann constant.

$$\Delta H = E - RT$$

$$\Delta S = R \left[\ln \left(\frac{hA}{kT} \right) \right] - 1$$

$$\Delta S \quad \Delta G = \Delta H - T\Delta S$$

Thermodynamic and kinetic data for specimens of PVC/BRP were listed in table 8 according to Coast-Redfern method.

Enthalpy of thermal decomposition also showed an increase in its magnitude this is because of heat evolution rate during thermal decomposition much higher than heat losing therefore heat accumulates in composites, however another reason also shares this phenomena is the low thermal conductivity of basalt, the negative sign of entropy means that the formed activated complex is higher organized than initial structure of composites and higher value of entropy higher thermal stability [30]. Figure 5 indicates that the slopes and intercepts of five straight lines were almost the same and it is known as kinetic compensation effect.

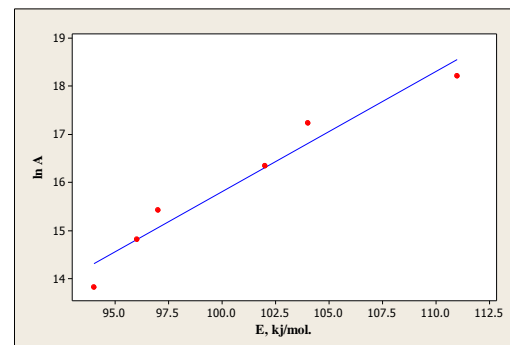


Fig.5. A plot of ln A versus E for thermal decomposition of PVC/BRP in Nitrogen flow

Figure.6 shows the DSC profiles obtained during thermal decomposition which showed the melting attitude of composites, table 9 shows the results obtained (melting of composites and its enthalpies) by DSC.

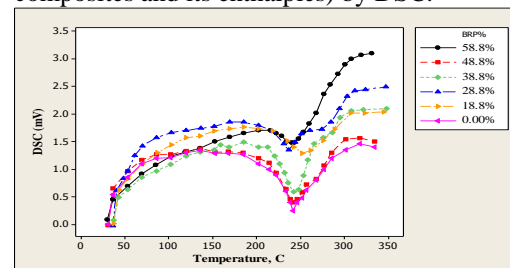


Fig 6 DSC profiles for thermal decomposition of PVC/BRP in nitrogen flow

Table 6 : The extent of thermal decomposition of PVC/BRP composites in nitrogen flow

BRP%	Weight percent loss on thermal decomposition						
	10%	15%	20%	25%	30%	35%	40%
	Temperature profiles, C°						
58.8	254.3	260.4	268.6	279.9	297.3	462.2	700.0
48.8	249.6	256.2	262.5	269.0	278.0	294.9	457.0
38.8	248.6	257.0	264.1	270.0	276.1	279.0	291.1
28.8	242.4	249.5	256.0	263.5	272.1	275.5	289.9
18.8	242.4	248.5	254.1	259.3	263.1	267.5	274.0
0.00	239.5	246.7	250.2	257.5	261.5	265.1	272.2

Table 7: Kinetic parameters of thermal decomposition of PVC/BRP composites

BRP%	Activation Energy, E (KJ/mol)	Reaction rate constant, A (S ⁻¹)	Peak Temperature, C°	R ²
58.8	110.493	8.2327x10 ⁷	(1) 260.0	98.0 %
			(2) 472.0	
48.8	103.509	3.0840x10 ⁷	(1) 225.0	97.5 %
			(2) 470.0	
38.8	101.680	1.2535x10 ⁷	(1) 260.0	97.1 %
			(2) 469.7	
28.8	96.608	0.5057x10 ⁷	(1) 253.0	99.1 %
			(2) 474.8	
18.8	96.192	0.2726x10 ⁷	(1) 273.6	98.9 %
			(2) 478.7	
0.00	94.54	0.1012x10 ⁷	(1) 273.5	98.6 %
			(2) 472.3	

Table 8: Thermodynamic properties of PVC/BPR composites

BRP%	ΔH , KJ/mol	$-\Delta S$, J/mol	ΔG , KJ/mol
58.8	106.061	106.521	162.836
48.8	99.077	114.606	160.162
38.8	97.289	122.169	161.795
28.8	92.234	129.606	160.407
18.8	91.818	134.745	162.694
0.00	89.471	131.891	161.860

Table 9: Enthalpy and melting points data by DSC for PVC/BRP composites

BRP%	Mass of sample, mg	H, Enthalpy of melting, J/g	Beginning melting point, C°	Middle melting point, C°	End melting point, C°
58.8	4.30	40.0520	224.1	242.1	281.4
48.8	4.30	56.5180	220.9	241.9	289.4
38.8	4.30	42.0249	229.5	253.2	287.4
28.8	4.20	8.72080	(1) 222.5	236.9	247.3
			(2) 261.4	266.0	271.6
18.8	5.10	27.0430	222.3	243.9	264.3
0.00	4.87	26.3401	217.8	239.1	262.6

Conclusions

From the present investigation the following conclusions can be drawn.

- Due to the presence of strong intermolecular interaction that is responsible for reducing the elasticity of composite and restriction of polymer chain movements, increasing the BRP loading causes a decrease in tensile strength and also in elongation at break.
- The data showed a significant improvement in hardness shore A values for the PVC composite specimens. The 58.8 % content exhibited the higher hardness value, while the 18.8 % content showed the lowest value.
- The compression strength value of the PVC– BRP composite specimens is increased by increasing the BRP filler loadings.
- There is a relative drop in water absorbability as the BRP filler content increased. This is because the increase in filler content has the effect of filling the free cavity interspaces between PVC polymer chains. This in turn will reduce the water particles penetration through those chains.
- Thermal conductivity values increased with increasing BRP filler loading.
- Thermal decomposition of PVC/BRP composites is investigated by TGA and DSC techniques. PVC/BRP composites degraded in two stages, random scission and decyclisation followed by char and volatilization. The activation energy and entropy were found to be higher for composites with high percentages of BRP, where BRP content increases the thermal stability. Negative sign of entropy indicates the activated complex is much more organized than the initial structure of the composites. The linear relationship between $\ln A$ versus E proves that the reaction model is correct. Moreover, DSC profile shows some decrease in the middle melting point for some specimens with decreasing of BRP contents.

Conflict of interest

The authors declare that they have no conflict of interest.

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