

Effect of Kaolinite on the Mechanical Properties, Thermal Properties, Flammability and Water Absorption Percentage of Poly (Vinyl Chloride) Composite

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Abstract

PVC/Kaolinite composites were prepared by the melt intercalation method. Mechanical properties, thermal properties, flammability and water absorption percentage of prepared samples were tested. Mechanical characteristic such as tensile strength, elongation at break; hardness and impact strength (charpy type) were measured for all samples. It was found that the tensile strength and elongation at break of PVC composites decreased with increasing kaolinite loading. Also, the hardness of the composites increases with increase in filler content. The impact strength of the composites at the beginning increases at lower kaolinite loadings is due to the lack of kaolin adhesion to the matrix. However, at higher kaolin loadings. This severe agglomeration of the filler due to its high surface energy creates many crack-initiation and stress-concentration sites, which are sensitive to impact stresses and cause detrimental effects on the impact properties of the composite. There is a general increase in absorption rate with increase in kaolin content. Thermal properties of PVC /kaolinite composites were characterized using Differential Scanning Calorimetry (DSC) and thermal conductivity analyzer. The results showed T_g shifted toward higher temperature for kaolinite composites compared to neat PVC. Also, thermal conductivity measurement value and effusivity increased with increasing filler loading. Heat capacity decreased with increasing filler content. The higher the filler content the higher burning time, the lower rate of burning and the lower height of the flame which are evident at 12wt. % for kaolinite.

Key words: Polymer, PVC/ Kaolinite Composites, Mechanical Properties, Thermal Properties.

Introduction

The polymer is a word of a Greek origin where “poly” is a synonym of “many”, while “meres” stands for “parts”, so a polymer is a large molecules consisting of repeated smaller size chemical units. They can be made into types of final products as it is the case with the pure (new) form.

Nonetheless, to make an effectively useful polymer, it is indispensable to modify it, particularly when the major limitations in the unmodified polymer are taken into account. Among those restrictions may be the low stiffness, low strength and the lack of stability when exposed to light rays, heat and

radiation that can ionize it. To make polymer vastly usable in different industrial fields nowadays, improved polymer composite productions are the right pathway to overcome the evident restrictions on one hand, and to insure the production of high quality polymers on the other [1].

A composite is defined as the combination at microscopic level between a couple (and more) of particular materials with a specific intervention to link them together. The material could be of a metal, ceramic or polymer nature [2].

Nowadays, poly vinyl chloride occupies the third position in the list of the most commonly produced plastics, where the polyethylene comes at the top followed by polypropylene, by virtue of its valuable characteristics, wide applications, barrier properties, low cost and high chemical resistance. PVC is in the form of powders, slurries, liquids, and tablets. It has a wide range of properties of colors, solid, rigid, and stiff materials (with high viscosity) at room temperature, light weight with a good resistance to bases and acids, alcohol, oils, compound hydrocarbon aliphatic. PVC has relatively low cost, biological and chemical resistance and workability and can be formed easily. For these reasons it is used for a wide range of applications, such as insulation on electrical cables, solid pipe manufacturing, window frames and doors, bottles, containers, furniture industry, etc. [3]

The thermal stability and processing of poly vinyl chloride, on the other hand, are unimportant in comparison with familiar polymers. These properties can be improved by making the poly vinyl chloride compound with some additives.

The most commonly used additives in PVC are lubricants, heat

stabilizers, plasticizers, fillers and pigments. Either polymerization or melt blending processes are used [4, 5].

The Kaolin is an ordinary phyllosilicate mineral, with a chemical formula $Al_2Si_2O_5(OH)_4$. This clay has 1:1 di-octahedral structure pattern consisting of tetrahedral SiO_4 and octahedral $Al_2(OH)_4$ plates. Specific layered consists of both of them which are neatly connected by the hydrogen bond between surface hydroxyl groups on the octahedral side and the basal oxygen atoms on the tetrahedral side. Kaolin finds a number of industrial usages; in making paints, paper, plastics, rubber and ceramics. Its largest viable usage as filler in PVC is in producing electrical cables or film anti blocking, tiles and so on [6, 7].

Dibasic lead stearates (DBLS), dibasic lead phosphate (DBLP) are used as the stabilizer; stearic acid is used as a lubricant. Dioctyl phthalate (DOP) is used as a plasticizer for compounding. It is noticed that raising the PVC temperature to more than 70 °C imposes some absurd changes on its properties. In practice, when temperatures of (150-200 °C) are used, sufficient degradation may occur upon normal processing operation which makes the product useless [8, 9].

In this research, the PVC/kaolinite composite preparation by the melt intercalation method is reported. In addition the influence of untreated kaolinite on the mechanical properties, thermal properties, flammability and water absorption percentage of poly vinyl chloride is investigated.

Experimental Work

1. Materials

The PVC powder, a suspension polymer, supplied by Saudi Basic Industries Corporation and Kingdom

of Saudi Arabia; The PVC used in this research has been analyzed by FTIR.

Iraqi kaolin powder $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ was brought from Ministry of Industry/ the State Company of Geological Survey and Mining. The specifications of the kaolin clay are given in Table 1. Additives such as: dibasic lead stearates (DBLS) ($2\text{PbO}\cdot\text{Pb}(\text{C}_{17}\text{H}_{35}\text{COO})_2$), dibasic lead phosphate (DBLP) ($\text{O}_8\text{P}_2\text{Pb}_3$) are used as the stabilizer; stearic acid ($\text{C}_{18}\text{H}_{36}\text{O}_2$) is used as lubricant. These materials are from the Ministry of Industry /Baghdad Plastic Plant. Dioctyl phthalate (DOP) (C_6H_4 -1, 2- $[\text{CO}_2 \text{CH}_2 \text{CH}(\text{C}_2\text{H}_5)(\text{CH}_2)_3\text{CH}_3]_2$) used in this study was used as a plasticizer by Indian Company/ Sigma Ultra.

Table 1: the characteristics of the Kaolin

component	Percentage%
SiO_2	47.14
Al_2O_3	34.74
Fe_2O_3	1.26
TiO_2	1.2
CaO	<1
Mg	0.25
SO_3	0.07
Moisture	14.44

2. Methods

• Sample Preparation

The following couple of steps summarize the production of poly vinyl chloride composite [10]:

In the first one, a 1% by weight of the stabilizer (Dibasic Lead stearate, DBLS and Dibasic Lead phosphate, DBLP) was added to the PVC powder. A 1% by weight of the stearic acid as a lubricant was also added. In addition to that, a percentage of 3%, 7% and 12% by weight of the filler were added. The filler used were bentonite and organoclay. The blend was mixed in a high_ speed mixer for 20 min forming a dry mixture.

In the second step, the dry blend was blended with 20% by weight dioctyl phthalate (DOP). The details of additives percentage are given in Table 2. The blending took place in the plastograph internal mixer (PLASTIC-CORDER), (Bra Bender .GmbH & CO .KG, Duisburg, Germany, 0-1000 $^{\circ}\text{C}$) at (65-80 $^{\circ}\text{C}$) for 20 min and a 64-rpm mixing speed.

Table 2: PVC composite formulation with filler Composition (%)

Material	1	2	3	4
PVC	100	100	100	100
Stabilizer :DBLS	1	1	1	1
Stabilizer :DBLP	1	1	1	1
Lubricant: Stearic acid	1	1	1	1
Plasticizer :DOP	20	20	20	20
kaolinite	0	3	7	12

• Compression molding (Hot press)

A hydraulic press Figure 1 (made in ENGLAN, NOORE, BIRMINGHAM and SERIAL NUMBER /D369) was used to mold the sheets to be tested in this study. It consisted of lower and upper moving platens. A sample of the above mentioned mold was wrapped by an aluminum thin sheet [1]. Using the controlled hydraulic ram, the lower platen was pushed toward the upper platen causing the mold to close and putting under pressure. The compression of the resulting film took place under the following conditions; a temperature of 170-185 $^{\circ}\text{C}$, and after pressure of 10 bars for 7 min. When the seven minutes, the pressure was removed letting the lower platen to fall down under the effect of gravity. The mold was dismantled rapidly and running water was used to cool it down. The aluminum foil paper was neatly un-wrapped to clear the produced film sample out of it. The compression-molded sheets of 90 gm weight and 180 * 180 mm size with 5 mm thickness were fabricated; this is agreement with [11].



Fig. 1: Hydraulic press equipment

Measurements

1. Mechanical Properties

• Tensile Strength and Elongation at Break

Mechanical Properties of the polymer blends films were measured on a Shimadzu Autograph in air at room temperature. Tensile strength and elongation at break point were measured using a universal testing machine (tension_ compression to 5KN; manufactured by Tinius Olsen (UK). The compressed sheets were cut into dumbbell-shaped specimens according to ASTM D647-68.

• Hardness

The hardness of all specimens was read and recorded; using a Vickers diamond indenter (Digital Micro hardness Tester), HVS- 1000. Vickers indentation is a valid tool for evaluating the hardness of polymers. With a load of 0.4403 N. The load is applied for time 20 s (ASTM Standard "Test Method for Vickers Hardness", 1997). For every specimen, three readings were taken in accordance with Polymeric Matrix Composite (PVC) ASTM-02240-97 for Hardness test. The hardness value was determined by the penetration of the Durometer indenter foot into the specimen. The

sample was placed on a flat surface on the pressure foot of the instrument. A calibrated spring in the Durometer applies a specific pressure to an indenter foot parallel to the surface of the surface of the specimen.

• Impact Strength

Impact test sample specimens were fabricated by the standard specification [ASTM-E23] and which is appropriate for testing by the impact device type Charpy (produced by (Tokyo Koki Seizosho, Ltd) company). The depth of the groove in the samples is 5mm with the groove base radius of 25 mm and a groove angle 45°. For the purpose of identifying the extent of impact strength which the composite material is capable of withstanding. From the Equation 1 we see that the impact strength value is calculated:

$$\text{Impact strength} = \frac{\text{Fraction Energy (KJ)}}{\text{Cross-Sectional Area (m}^2\text{)}} \dots(1)$$

• Water Absorption of the Composites Samples

Water absorption test was performed according to ASTM D570-99 (Standard Test Method for Water Absorption of Plastics, 1998) by conditioning and weighing the sample. Each sample was immersed in distilled water in a transparent thermoplastic container and covered with the lid for 24 hr at 27°C. Excess water on the sample surface was wiped off with a filter paper before reweighing Turu et al. [1]. From Equation 2 we see that the percentage increase in mass during immersion was calculated using the equation:

$$\% \text{ Water Absorption} = [(\text{Wet weight} - \text{Dry weight}) / \text{Dry weight}] * 100 \dots(2)$$

Wet weight: is the weight of sample after immersing in water for 24 h (g)

Dry weight: is the initial weight of sample (g).

2. Thermal Properties

• Differential Scanning Calorimeter (DSC)

The thermal properties of the composites were determined by Differential Scanning Calorimeter DSC measurements performed using a Shimadzu DSC-50, Japan, at a heating rate of 10°C/min in the temperature range (25-250) °C under nitrogen purge. The glass transition temperature was taken as the midpoint of the transition.

• Conductivity Measurement (k, ε and Cp)

Thermal Conductivity measurements were done using SETARAM, Instrumentation, KEP Technologies-THERM TCi™, and Thermal Conductivity Analyzer/Made in France. The Mathis TCi is based on the modified transient plane source technique. It uses a one-sided, interfacial, heat reflectance sensor that applies a momentary, constant heat source to the sample. Both thermal conductivity and effusivity are measured directly and rapidly, providing a detailed overview of the thermal characteristics of the sample material. Sample material can be a solid, liquid, paste or powder [12, 13]. From the Equation 3 we see that the heat capacity values of polymer composite [14]:

The effusivity equation by ($Ws^{1/2} / m2 .K$)

$$\epsilon, \text{effusivity} = \sqrt{k \cdot \rho \cdot Cp} \quad \dots(3)$$

3. Flammability

• Burning Speed Measurement Test [To measure the time required for burning until a full self-extinguish take place using (ASTM: D_635)]

This method is considered as an internationally adopted one and is used to measure the flame dispersing speed in the different polymer material and to calculate the time duration needed for the burning. It is vastly used in polymer material convertible into plates, sheets or bars. Polymers test able in this method are categorized to be flammable and self-extinguishable after a certain time of burning according to ASTM: D_635.

• Flame Height Measuring Using the Test (ASTM: D-3014)

This test is one of the lab methods used to measure the flame height a burning polymer can make. It is vastly used in polymer material convertible in to plates, sheets or bars.

Results and Discussion

From the data presented in Table 3, Figure 2 and 3 it can be seen that the increase in filler content causes a decrease in tensile strength which can be attributed to the decrease in the interfacial forces between PVC crosslinks across the interface and the filler particles. Also, a decrease in elongation at break is noticed. This may be due to the fact that the molecules of the fillers can distribute in the inter-aggregate space by an inter-structural process according to Dimitry et al. [15]. Likewise, the decrease in tensile strength and elongation at break with increasing filler content can also be attributed to the weak points in the material caused by the aggregates. These can break very easily by the applied stress .When broken, the aggregates concentrate the stress strongly thus decreasing the tensile strength of kaolin. This is in

agreement of the results of Leong et al. [16].

Table 3 gives the test results for the hardness values for the basic PVC material reinforced with kaolin and Figure 4; the results show a significant

improvement in the values of hardness for the different content of reinforcement. The 12% content showed the best hardness values, this is agreement with [4].

Table 3: Mechanical Properties of Unfilled PVC, PVC-kaolinite composites

Sample Designation	Tensile Strength (MPa)	Elongation%	Shore A Hardness	Impact strength (kJ/m ²)
PVC neat	2.615	45.6	8.7	4.57
PVC-3% kaolin	1.499	4.613	16.5	9.472
PVC-7% kaolin	1.259	2.533	17.6	6.06
PVC-12%kaolin	0.907	0.567	30	4.12

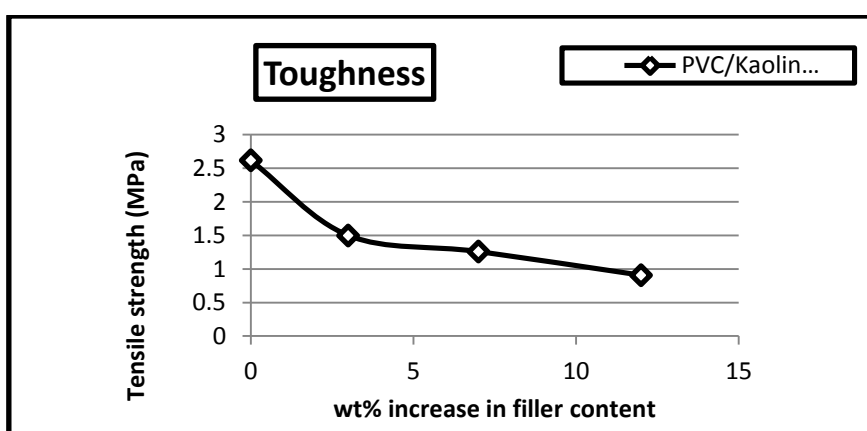


Fig. 2: Tensile strength curve of PVC /kaolinite composite

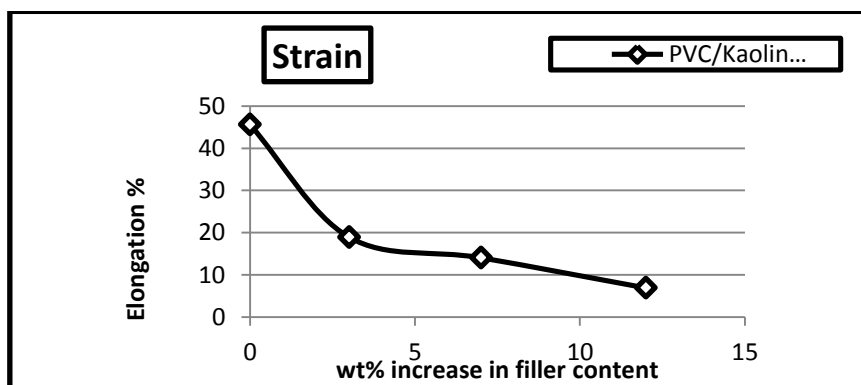


Fig. 3: Elongation curve of PVC/kaolinite composite

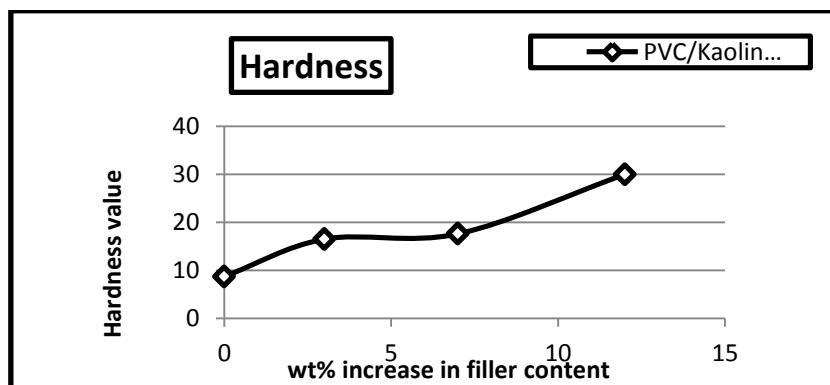


Fig. 4: Hardness (Shore A) of Unfilled PVC and PVC/ kaolinite composite

Table 3 and Figure 5 illustrate how the impact strength value changes with the change of the content added of kaolinite. Despite the good dispersion of the filler, the lack of kaolin adhesion to the matrix can lead to the impact strength values. When the loading of kaolin is high, its surface energy will be high causing severe agglomerates of filler. This can initiate many cracks and many stressed sites, which are susceptible to stress impacts, causing damaging effects on the impact properties of the composite [16]. When the kaolin is added to the PVC with a weight content of 3%wt and 7%wt, an increase on the water absorbability of the PVC occurs in Figure 7, Table 4. This behavior might be due to the presence of void spaces in the matrix which could have been formed during compounding of

composites. The void spaces in the matrix accommodates the water absorbed [1]. Kaolinite which is hydrophilic which is inorganic materials have the effect of absorbing water particles which contributes to the increase of samples' weight. When 12%wt clay content is added a relative drop on the absorbability is noticed compared with the other content. This is attributed to the fact that the increases on kaolin content have the effect of filling the free interspaces between polymer chains which reduces the penetration of water particles through those chains. This proves that using this particular content reduces the water absorbability of the PVC retaining at the same time the improvement of mechanical properties such as hardness.

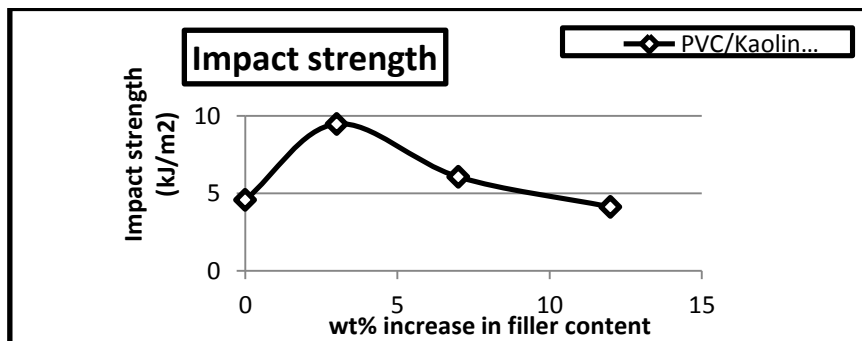


Fig. 5: Impact Strength curve of Unfilled PVC and PVC/ kaolinite composites

Table 4: Water Absorption percentage Results for PVC/ Kaolin Composite for 5 day

% Kaolin	% water uptake(after 24 h of immersion at 25 ⁰ C)				
	%day1	%day2	%day3	%day4	%day5
0	0.722	1.08	1.805	4.33	5.05
3	1.68	2.70	3.71	6.75	8.10
7	2.08	2.68	3.88	7.16	8.05
12	1.15	1.38	1.48	2.07	2.53

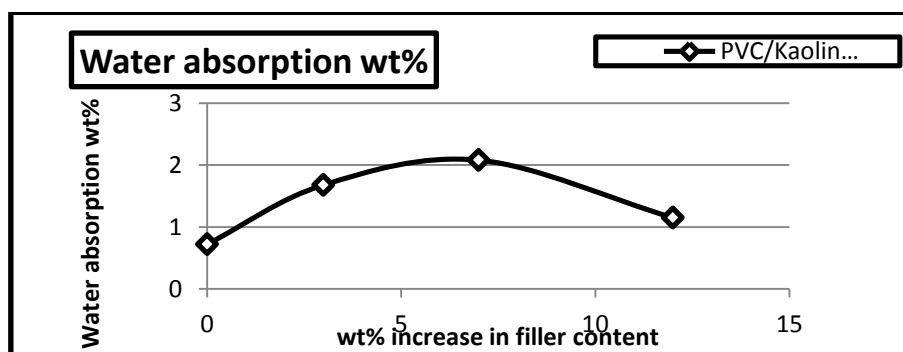


Fig. 6: Plots water content against percentage increase in filler (after 24 h of immersion at 25⁰C)

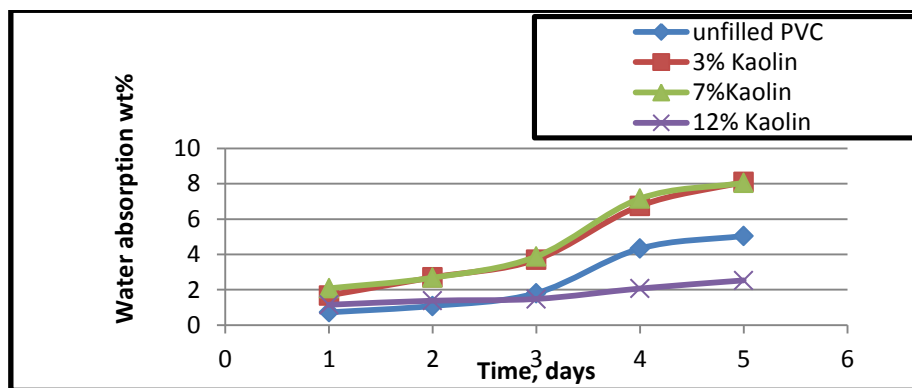


Fig. 7: Water absorption percentage of unfilled PVC, PVC/kaolin composite versus time

Based on the data collected from the DSC thermo-gram Table 5 and Figure 8, 9 and the glass transition temperature were found out. Whereas the fillers fill and the gaps or spaces between the polymers chain, they

restrict the chains movement and improve their plastic properties. The results showed $T_{3\%}$, $T_{7\%}$ and $T_{12\%}$ shifted toward higher temperatures for kaolinite compared to neat PVC. This is agreement with [17, 18].

Table 5: Thermal Properties of Unfilled PVC, PVC-kaolinite composites

Sample Designation	Glass Transition Temp. (T_g) ($^{\circ}\text{C}$)	Thermal conductivity value (k) (W/m. K)	Effusivity(ϵ) ($\text{Ws}^{1/2} / \text{m}^2 .\text{K}$)	Heat capacity Results cp (J/g.K)
PVC neat	89.09	0.222	580.4	1083
PVC-3% kaolinite	98.70	0.240	601.1	1052
PVC-7% kaolinite	100.47	0.291	657.5	1010
PVC-12% kaolinite	103.37	0.437	812.3	986.8

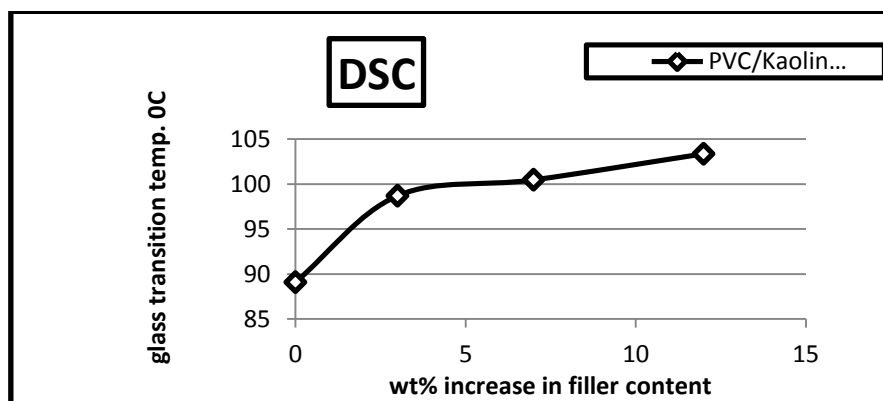


Fig. 8: Plots Glass Transition Temperature against percentage increase in filler

As the content of the kaolinite filler increases so does the thermal conductivity Figure 10, accompanied by the increase of thermal effusivity Figure 11, and a decrease in heat capacity Figure 12, This is agreement with [14].

The results of the measurement showed a large increase in the burning time and showed large decrease in the rate of burning for PVC/kaolinite composite, with other words the higher

the kaolin content the higher burning time and the lower rate of burning which are evident in Figures 13 and 14, Table 6 , This is agreement with [19].

The results of the measurement showed decrease in the flame height (H) for PVC/kaolinite composite. This decrease in the flame height (H) is inversely proportional with the increase of the kaolinite content to the neat PVC with other words the higher

the filler content the lower rate of the flame height (H) which is evident in

Figure 14 and Table 6, This is agreement with [20].

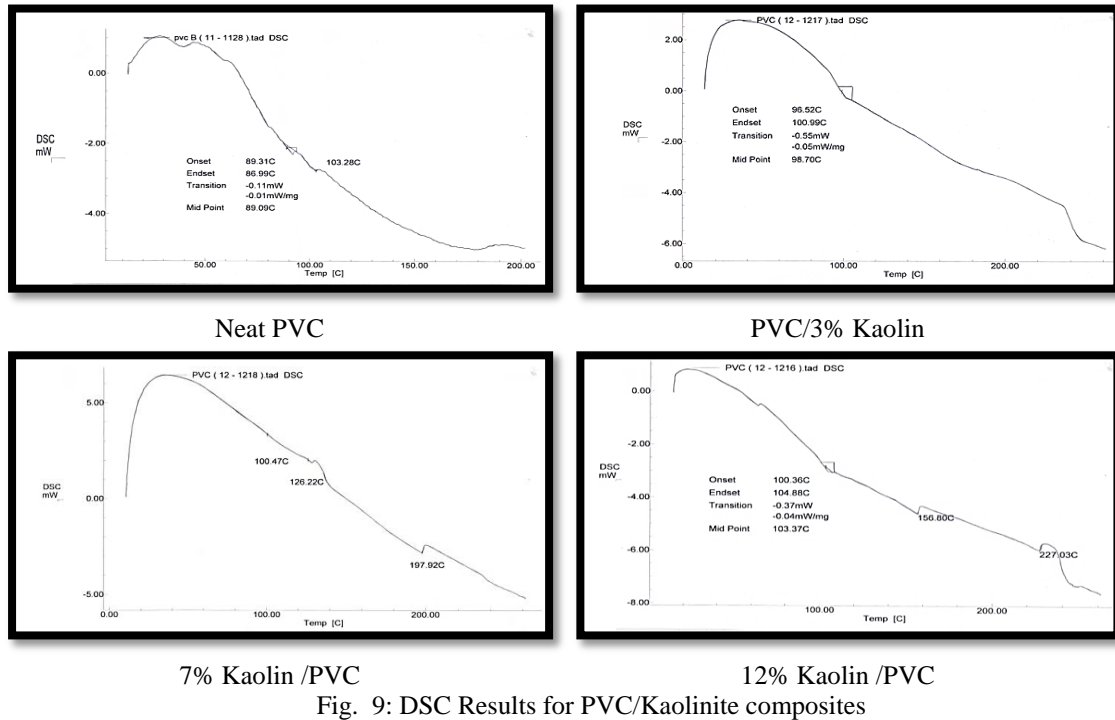


Fig. 9: DSC Results for PVC/Kaolinite composites

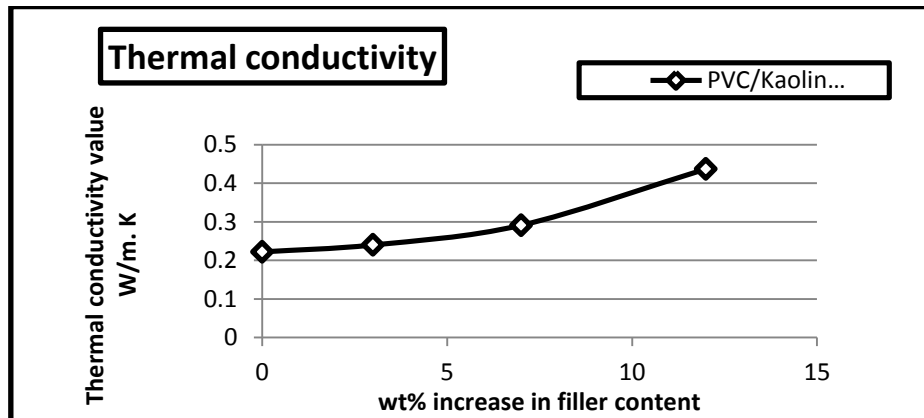


Fig. 10: Thermal conductivity against filler content

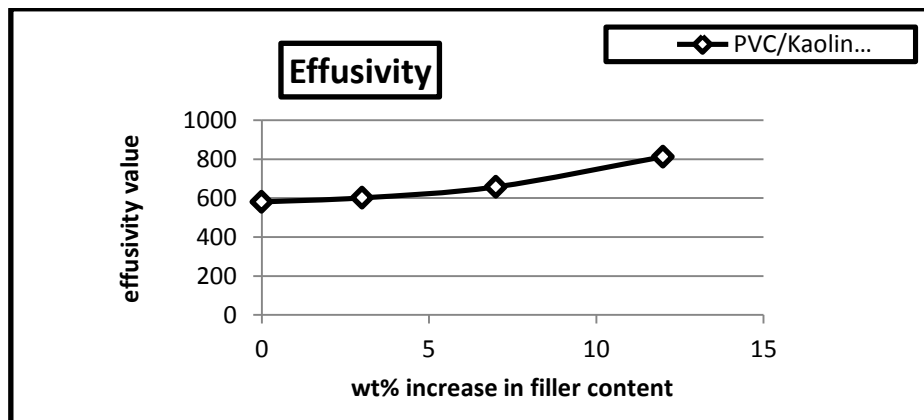


Fig. 11: Effusivity against filler content

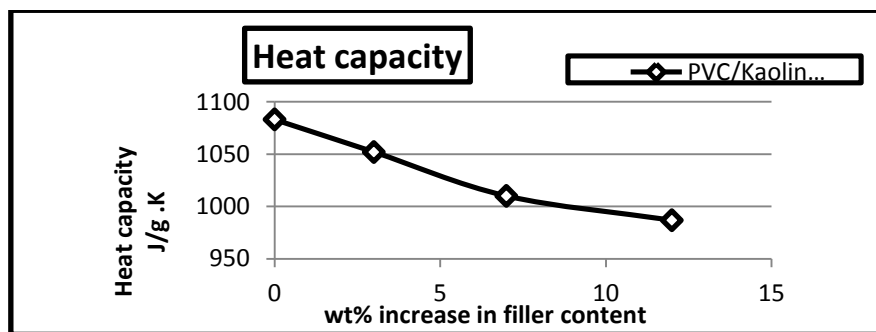


Fig. 12: Heat capacity against filler content

Table 6: Flammability Properties of Unfilled PVC, PVC-bentonite composites and PVC-Organoclay composites

Sample Designation	Burning Time (min)	Rate of Burning (cm/s)	Height of Flame (mm)
PVC neat	6	0.027	50
PVC-3% kaolinite	11	0.015	18
PVC-7% kaolinite	14	0.011	15
PVC-12% kaolinite	20	0.0083	12.5

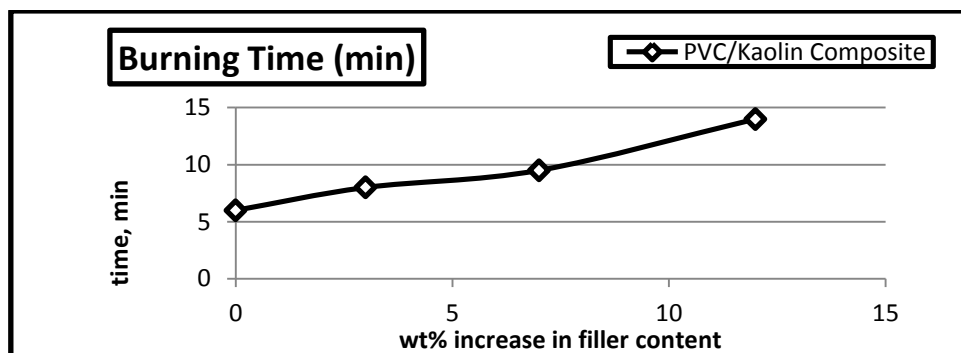


Fig. 13: The burning time against filler content

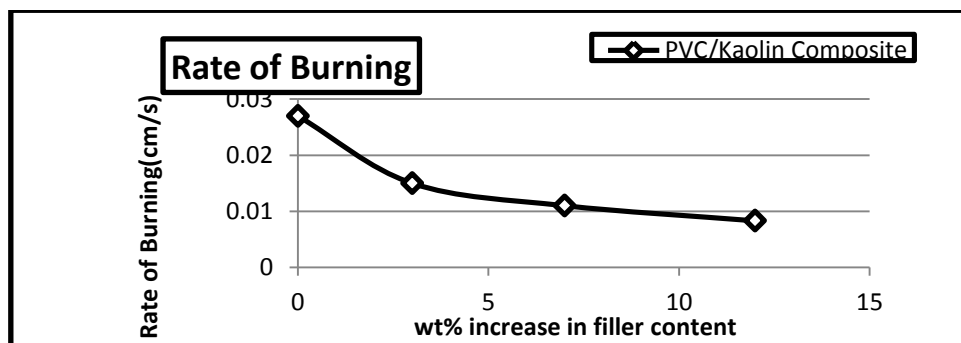


Fig. 14: Rate of Burning against filler content

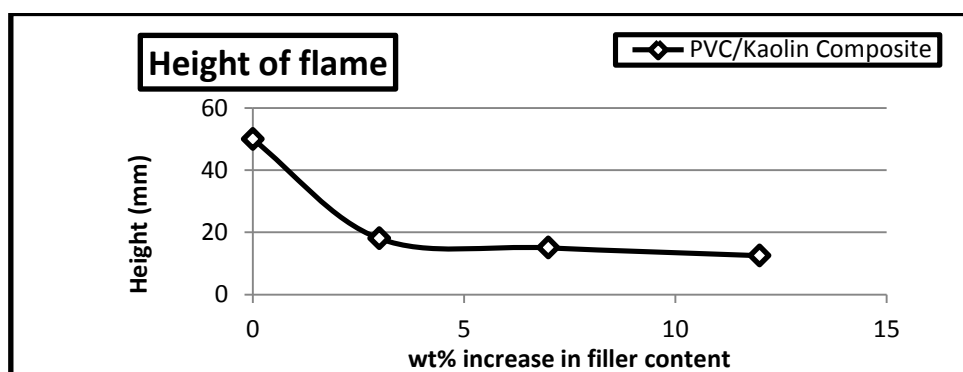


Fig. 15: Flame height against filler content

Conclusion

By analyzing the results obtained in this work, the following conclusions can be drawn:

1. Increasing the kaolinite loading causes a decrease in tensile strength which can be attributed to the decrease in the interfacial forces between PVC crosslinks across the interface and the filler particles. Also, a decrease in elongation at break is noticed. This may be due to that the molecules of the fillers can distribute in the inter-aggregate space by an inter-structural process.
2. The results showed a significant improvement on the values of hardness for the different content of reinforcements. The 12% content showed the best hardness values.
3. It is noticed that the impact strength value of the kaolin at the beginning increase the impact resistance at lower kaolin loadings. This is due to the lack of kaolin adhesion to the matrix, although there is good dispersion of the filler. However, at higher kaolin loadings, severe agglomeration of the filler due to its high surface energy creates many crack-initiation and stress-concentration sites, which are very sensitive to impact stresses and cause detrimental effects on the impact properties of the composite.
4. When 12% wt clay content is added a relative drop in the absorbability is noticed compared with the other content. This is attributed to the fact that the increase in kaolin content has the effect of filling the free interspaces between polymer chains which reduces the penetration of water particles through those chains. This proves that using this particular content reduces the water absorbability of the PVC.
5. The results showed $T_{3\%}$, $T_{7\%}$ and $T_{12\%}$ (T_g) shifted toward higher

temperatures for kaolinite compared to neat PVC.

6. Thermal conductivity values and effusivity increased with increasing filler loading. Heat capacity decreased with increasing filler content.
7. The higher the filler content the higher burning time, the lower rate of burning and the lower height of the flame which are evident at 12wt%.

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