

The Influence of Treating Low-grade Kaolin on Mechanical, Thermal and Electrical properties of the PVC/Kaolin Composite: An Example from Sinai, Egypt

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ABSTRACT

The present study aims to investigate the role of raw and treated kaolin as a filler in the main characteristics of Poly Vinyl Chloride (PVC) composites. Raw low-grade kaolin sample from the Sinai Peninsula in Egypt was physically and thermally treated using magnetic separation and calcination process at 700°C for 120 minutes, respectively, to produce calcined kaolin. Analysis by X-ray diffraction and X-ray fluorescence were performed on both untreated and calcined kaolin samples, which were used as fillers in the synthesis of PVC compounds. To characterize the resultant products, density, tensile strength, elongation, thermal stability, and volume resistivity tests were carried out on these components. The results show an improvement in the quality of the raw kaolin after the application of the beneficiation processes. The TiO₂% decreased from 3.72% to 2.78%, Fe₂O₃% decreased from 1.6% to 1.04%, and Loss on Ignition (LOI) decreased from 10.69% to 0.43%. Moreover, the calcined kaolin enhanced the tensile properties of the prepared PVC composites. The volume resistivity of the PVC composites were also increased from $2.1469 \times 10^{12} \Omega\text{cm}$ for PVC/raw Kaolin (KL) composite sample to $8.8870 \times 10^{13} \Omega\text{cm}$ for PVC/Calcined Kaolin (CKL). However, the thermal properties show higher thermal stability values for PVC/ KL samples.

Keywords: Calcined Kaolin; Electrical properties; Filler; Kaolin; PVC composites; Sinai peninsula

INTRODUCTION

The plastic industries occupy a very important role in the international economy in the latest years. Poly Vinyl Chloride (PVC) takes a leading position in the list of the most commonly produced plastics [1]. In fact, the PVC cannot be processed on its own due to its very low thermal stability and high melt viscosity [2]. Therefore, PVC is necessarily combined with a number of essential additives such as plasticizers, fillers, and stabilizers to give a varied range of properties and to satisfy several demands [3-4]. The fillers are solid particulates or fibrous materials that are inert chemically. They are used in PVC to modify the mechanical properties and to reduce the cost of products [1]. The worldwide most common filler is calcium carbonate. Fillers such as Aluminum Tri-Hydroxide (ATH), talc, dolomite, and clay minerals are used as functional fillers to improve particular property of the PVC composites [5].

Kaolinite is the main mineral in the kaolin group, which composed of hydrated aluminum silicate with the chemical formula $(\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_2)$ [6]. Kaolin is one of the most important economic materials

used in many industrial applications such as ceramics, glass, and plastics due to its unique chemical and physical properties [7-8]. It is processed by mechanical methods such as crushing, grinding, and screening; however, it is necessary to use other chemical and mechanical processing such as drying, thermal calcinations, surface treatment and extruding, to prepare it for industrial usages [9-11].

Several researches have been published over the last two decades on the effect of modified kaolin in PVC composites [12]. Studied the Silane-modified kaolin as a filler in the physical, mechanical, and structural properties of PVC composites [13]. Prepared PVC/kaolin nano-composites in the solid-state using a pan milling method [14]. Prepared novel solvent-free kaolin fluid to improve the mechanical and thermal properties of PVC composites and compared with original raw kaolin [15]. Synthesized coated kaolin using liquid macromolecular modifier, then studied the thermal and mechanical properties of the as-prepared Rigid PVC/coated kaolin [15]. The present study; however, aims to investigate the effect of Egyptian raw as well as treated kaolin on the mechanical properties of PVC composites such as tensile strength, elongation

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Figure 1: Location of the lower cretaceous sedimentary kaolin deposits in musabaa salama mountain in sinai peninsula.

Table 1: Physical and chemical properties of pvc resin, di-octyl phthalate and ca/zn stability.

Materials	Physical and Chemical properties	
	Property	Results
PVC (k-67)	Color	White
	Specific gravity	1.4
	Apparent Bulk Density (g/cm ³)	0.49-0.51
	Porosity (DOP) (ml/gm)	0.2-0.32
	Mass Loss Wt %	0.3 max
DOP ^a	Appearance	Colorless liquid
	Odor	Slight
	Specific gravity	0.984 at 20°C
	Freezing point	
	Boiling point	385°C
	Flash point	206°C
	Volume Resistivity	2 × 10 ¹¹ Ω ^b .cm
Ca based Stabilizer	Color	White
	Appearance	Powder
	Ash (% at 800°C)	48
	Bulk Density (g/L)	450 ± 10%

^a DOP (DI-Octyl phthalate) plasticizer

^b (ohm) Electrical resistivity unit

Table 2: The Formulations of PVC/raw kaolin (PVC/KL) composite samples and PVC/calcined kaolin (PVC/CKL) composite samples in Part per Hundred resins (phr).

Material	PVC Resin (k-67)	Filler		DOP Plasticizer	Stabilizer (Ca/Zn based stabilizer)
		Raw kaolin	Calcined kaolin		
PVC - free from fillers	100	-	-	50	3
PVC/15 KL ^a	100	15	-	50	3
PVC/30KL	100	30	-	50	3
PVC/45 KL	100	45	-	50	3
PVC/15 CKL ^b	100	-	15	50	3
PVC/30CKL	100	-	30	50	3
PVC/45CKL	100	-	45	50	3

^a Raw kaolin material

^b Calcined kaolin material

at break, and density. Thermal stability and volume resistivity were also applied. The latter is to indicate the electrical properties.

MATERIALS AND METHODS

The kaolin sample was collected from the lower Cretaceous Malha Formation in Musabaa Salama Mountain area at Latitude: 29°6'21" and Longitude: 33°14'12" in the Sinai Peninsula (Figure 1) in the northeastern part of Egypt [16-18].

Treatments of Kaolin (KL) in this study was applied by two processes, the first process was the physical treatment of kaolin by magnetic separation method to decrease the coloring agents such as iron oxide Fe_2O_3 and titanium oxide TiO_2 . The second process was the thermal treatment of kaolin by heating for about two hours at temperature about 700 °C to remove hydroxyl content (OH^-). Consequently, the kaolinite mineral in kaolin will be converted into meta-kaolinite and this process is known as calcination process [7,19,20]. The resultant kaolin after treatments is referred to as "Calcined Kaolin" (CKL). (Table 1) shows the physical properties of the chemical stabilizers used in the PVC.

Preparation of treated kaolin

Crushing and grinding: The sample was crushed in a closed-circuit using a Denver jaw crusher. After that, about 3 kilograms of the kaolin was introduced to the grinding process (Attrite-Mill Union machine) to produce a very fine-grained raw kaolin powder.

Physical treatment of kaolin: About 2 kilograms of the raw kaolin was subjected to magnetic separation process using the wet high-intensity magnetic separator at the maximum magnetic field in order to pick up the weakly magnetic particles. The magnetic separation was performed at a matrix loading capacity of 2.5% and a retention time of 2.5 minutes at a feeding rate of about 240 ml/min.

Thermal treatment of kaolin: After magnetic separation, about 1.5 kilograms of the resulted material was heated in a laboratory furnace at 700°C for 120 minutes [21]. Thereafter, the sample was cooled to room temperature at ambient conditions to form meta-kaolinite (calcined kaolin). The estimated value for the removed hydroxyl content of raw kaolin (the degree of dehydroxylation D_{tg} of the kaolin sample) was estimated by calculating the ratio between the percentage of removed content from LOI_{AC} (loss of ignition after calcination) to the percentage of LOI_{BC} (loss of ignition before calcination) [22].

Preparation of PVC Composites

A standard PVC composite sample was prepared firstly without a filler as PVC natural insulation compound according to both 60502-1 and 50363-3 standards for insulating PVC compounds [23-24]. The sample was prepared by heating of PVC Resin in 100 per hundred resin (phr), Di-octyl Phthalate (DOP) as plasticizer in 50 phr and Ca/Zn-based stabilizer in 3 phr. Kaolin or calcined kaolin was added as a filler in 15, 30, and 45 phr, respectively Table 2.

The test sample was mixed in Benchtop Lab Mixer (make: Lab-Tech) at 2000 rotation per minute (rpm) till the complete formation of a dry blend of PVC insulation compound. The dry blends were mixed in a double roll mill machine at 135°C to form homogenous long sheets. Sample of the resulted long sheets was subjected to heat in compression press machine (model no. 6707- make: CEAST) at 193.3°C for 4 minutes to prepare test sheets of PVC composite with 12 cm length, 12 cm width, and 0.1 cm thickness. The composite test sheets for PVC/KL and PVC/CKL are shown in (Figure 2).

Characterization methods of kaolin

X-ray fluorescence analysis: XRF analysis was established for kaolin and calcined kaolin using wavelength dispersive X-ray Fluorescence spectrometry instrument in the Egyptian Mineral Recourses Authority (EMRA), to determine the chemical composition of the samples.

X-ray diffraction analysis: The mineralogical composition of raw kaolin and calcined kaolin were determined using X-ray Diffraction (XRD) Analysis. P-Analytical X-ray Diffraction equipment model X pert Pro with a Monochromator, Cu-radiation ($\lambda=1.542\text{\AA}$) at 50 K.V., 40 M.A, and Scanning Speed 0.02 O/sec were used.

Characterization techniques of PVC composites

Characterization of mechanical properties: Tensile strength and Elongation at break test is a significant test, which indicates the mechanical properties of the plastics. Test samples were prepared according to D 638 standard methods for Tensile and Elongation of plastics [25]. The test was performed using a computerized universal tensile machine (model no. N111201). Results were estimated at (Newton/mm) of tensile strength.

Density test is another significant mechanical test. The density of samples was estimated in gram per cubic centimeters (g/cm^3), and

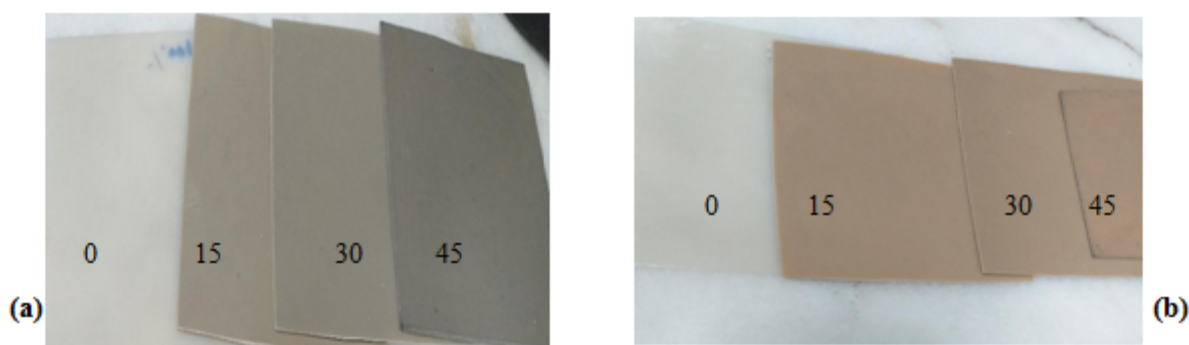


Figure 2: PVC composite test sheets with various content of fillers in 0 phr for standard free filler sample, 15, 30, and 45 grams of filler (a): PVC/raw kaolin composite test sheets; (b): PVC/calcined kaolin composite test sheets.

the test was performed according to the D792 standard method for density of plastics using AG balance meter (model no.1119121150 - make: Mettler Toledo) [26]. Sheeted samples with dimensions of $1 \times 1 \times 0.1$ cm were used as test specimens at temperatures 20-25.

Characterization of Thermal properties: A thermal stability test was carried out according to the (International standard method for Thermal Stability test) [27]. About 0.05 gram was inserted into a small glass tube (inner diameter equals 4.0 ± 0.5 mm). A pH paper with a pH range of 1 to 10 was inserted into the top of the glass tube as an indicator of the effervescence of colorless chloride atoms of polyvinyl chloride. The glass tube was inserted into a thermal stability Meter (model no. 100-make GP) at 200 ± 0.5 °C. The thermal stability of the sample was estimated by calculating the duration time since inserting the glass tube to the instrument until the appearance of the faint blue color using a suitable time meter.

Characterization of Electrical properties: The volume resistivity test was carried out according to the D257 standard method for the insulation resistance of materials, to evaluate the electrical properties of PVC compound [28]. The sample sheets were

Table 3: The X-ray fluorescence chemical analysis data (in wt%) of the Egyptian kaolin and calcined Egyptian kaolin.

Item	Raw kaolin	Calcined kaolin
SiO ₂	53.15	62.18
TiO ₂	3.72	2.78
Al ₂ O ₃	30.16	31.67
Fe ₂ O ₃	1.62	1.04
MnO	0.01	<0.01
MgO	0.07	0.24
CaO	0.08	0.23
Na ₂ O	0.08	0.03
K ₂ O	0.08	0.11
P ₂ O ₅	0.01	0.01
Cl	0.01	0.01
SO ₃	0.01	0.01
LOI	10.69	0.43
Total	99.69	98.74

immersed in water for 12 hours at a temperature of 20 ± 5 °C, then the sample sheet was dried then inserted between the electrodes of high resistance meter (model no. 4339 B-make: Agilent).

RESULTS AND DISCUSSION

Characterization of kaolin

The results of the XRF (Table 3 and Figure 3) show that The TiO₂ % decreased from 3.72% to 2.78%, Fe₂O₃ % decreased from 1.6% to 1.04%, and Loss on Ignition (LOI) decreased from 10.69% in raw kaolin to 0.43% in treated kaolin.

In contrast, SiO₂ and Al₂O₃ percentages increased from 53.15%, 30.16% in raw kaolin to 62.2%, 31.7% in calcined kaolin, respectively. This indicates that the physical treatment for raw kaolin, by magnetic separation method, caused a notable decrease in TiO₂ and Fe₂O₃ percentages, and the thermal treatment by the calcination process produced a remarkable decrease in loss of ignition content (LOI). Therefore, SiO₂ and Al₂O₃ percentages increased on the expense of the loss of other components during the calcination process. The decreasing of the coloring components such as titanium oxides and iron oxides enhances the brightness and whiteness of the kaolin [8]. As a consequence, the shape of the as-prepared PVC composites shows moderate improvements in the whiteness of PVC/calcined kaolin (PVC/CKL) samples than PVC/raw kaolin (PVC/KL) samples (Figure 2). Subsequently, the enhancement of the magnetic separation process and/or its repeatability could improve the whiteness and brightness of the raw kaolin. On the other hand, the particular decrease of the LOI from 10.7% in the raw kaolin to 0.43% in the calcined kaolin indicates that the degree of dehydroxylation (D_{tg}) of the kaolin sample equals about 0.96, which refer to the nearly complete dehydroxylation after the calcination process [21,22].

The X-ray diffraction results show that the raw kaolin sample is composed mainly of kaolinite mineral ($Al_2Si_2O_5(OH)_4$) and quartz (SiO₂). The peak of the kaolinite was observed clearly as shown in the Semi-quantitative analysis of the XRD chart (Figure 4). The peak of the kaolinite shows a significant decrement after calcinations (Figure 4). [29]. Claimed that the peak of kaolinite declines

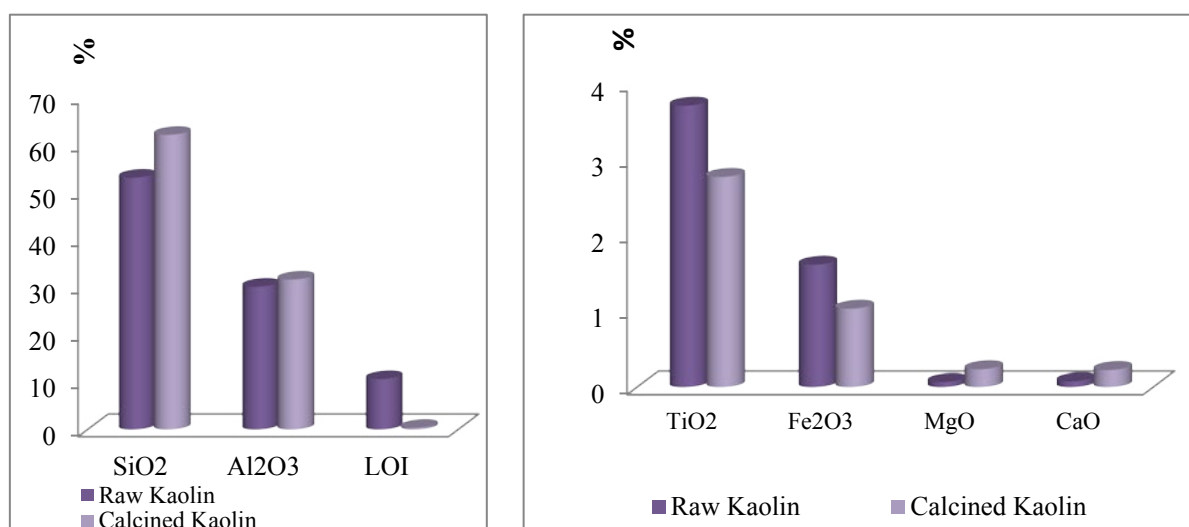
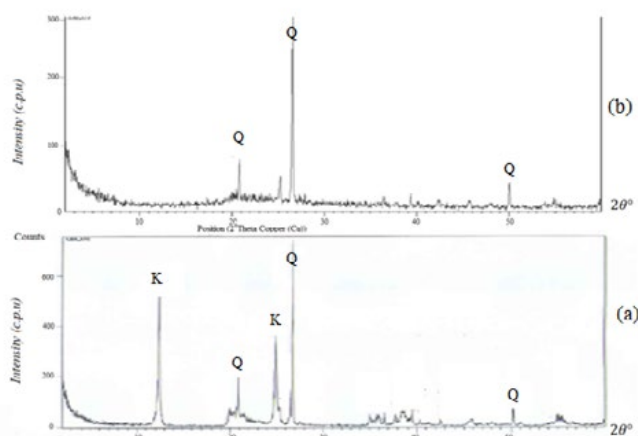
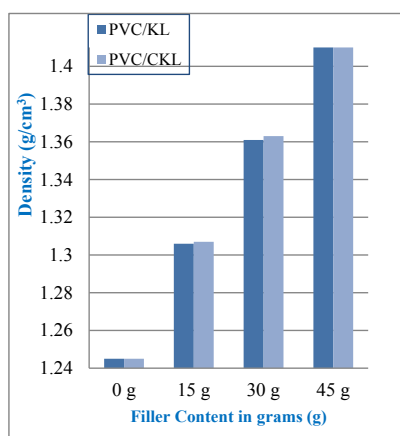


Figure 3: The chemical composition of raw kaolin and calcined kaolin by x-ray fluorescence analysis (a) Major constituents; (b): Minor constituents.

Table 4: Results from tensile strength, elongation at break density, thermal stability and electrical volume resistivity tests for PVC/raw kaolin (PVC/KL) composite samples and PVC/calcined kaolin (PVC/CKL) composite sample.

Material	Tensile Strength (N ^o /mm ²)	Elongation (%)	Thermal Stability (Minute)	Specific Gravity (g/cc)	Electrical Volume Resistivity (Ω/cm) At 500 volts
PVC-free from fillers	22.2	521	49	1.245	9.06E+11
PVC/15 KL	17.2	361	54	1.306	1.09E+12
PVC/30KL	14.6	275	59	1.361	1.55E+12
PVC/45 KL	12.9	231	63	1.41	2.15E+12
PVC/15 CKL	18.4	392	34	1.307	2.35E+13
PVC/30CKL	15.7	301	45	1.363	4.5E+13
PVC/45CKL	13.9	268	52	1.412	8.89E+13

*Newton per square centimeter (tensile strength unit)

**Figure 4:** XRD chart of kaolin samples (a): raw kaolin sample; (b): calcined kaolin sample.**Figure 5:** The Density of PVC/raw kaolin composites (PVC/KL) and PVC/ calcined kaolin composites (PVC/CKL).

evidently after the calcination process since the crystalline structure of kaolinite crumble and become amorphous dehydroxylated meta-kaolinite.

Characterization of PVC composites

The mechanical properties of PVC composites Density results of PVC/KL and PVC/CKL composites (Table 4 and Figure 5) show that the total density values (ρ_{total}) of PVC/filler composites increase with increasing the filler content in PVC composite, which indicates that both the kaolin and the calcined kaolin have

higher density values (ρ_{filler}) than the density of the PVC standard sample (ρ_{Standard}).

The densities of both kaolin and calcined kaolin could be calculated (Eq.1) as follow [30].

$$m_t = m_{st} + m_f = \rho_{st} V_{st} + \rho_f V_f \quad (\text{Eq. 1})$$

Where m_t is the total mass, m_t equals the sum of the mass of standard sample (m_{st}) and of filler in the sample (m_f) in grams; ρ_{st} and ρ_f are the density values of standard sample and filler respectively in gram/cm³; both (V_{st}) and (V_f) are the volume of total sample, standard sample and filler respectively in cubic centimeter.

Using the mass value (Table 2) and the density values (Figure 5), the density of both kaolin and calcined kaolin was estimated to be 2.59 gm/cm³ and 2.63 gm/cm³, respectively (Table 5). The slight increase in the density of kaolin after physical and thermal treatment can be attributed to the decrease in (OH)⁻ content after the calcinations process. Therefore, the heavy oxides percentages comparatively increased through the produced kaolin sample, and consequently the resulted increase in the density of calcined kaolin [31].

Calcium carbonate is considered as a global commonest filler. Its specific gravity was estimated as 2.7-2.75 [1,5]. The calcium carbonate and kaolin are accepted as cheap mineral fillers; however, the lower density of kaolin gives a higher chance to decrease the cost of the expensive PVC resin in the PVC composite. On the other hand, calcium carbonate has better whiteness and brightness properties, which gives calcium carbonate filler a higher priority only in the case of white-colored PVC composites.

Tensile strength and elongation test results show that when tensile strength was estimated for the standard free filler-PVC composite sample. Show that, it equal to 22.2 Newton per square millimeter (N/mm²) (Table 4 and Figure 6).

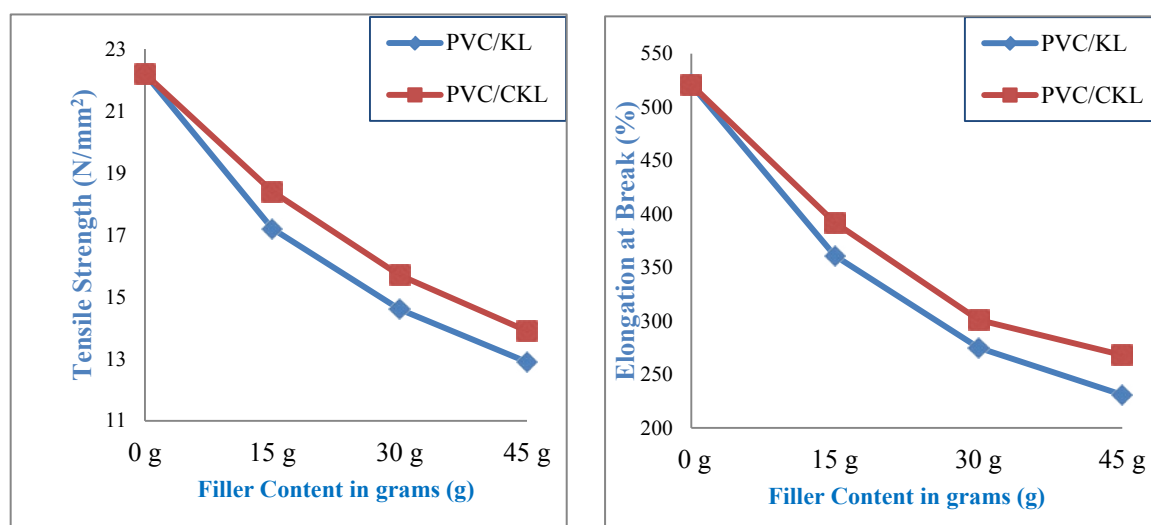
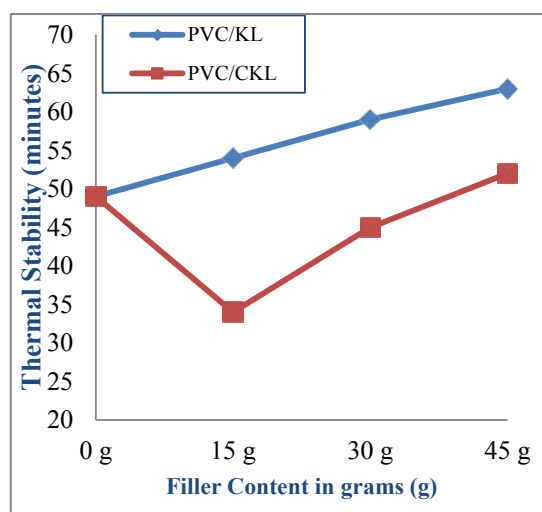
This value decreased gradually with increasing raw kaolin and calcined kaolin fillers. The PVC/calcined kaolin composite sample has tensile strength values of 18.4 N/mm², 15.7 N/mm², and 13.9 N/mm² for 15 phr, 30 phr, and 45 phr of PVC/calcined, respectively. Contrariwise, the corresponding PVC/raw kaolin composites have values of 17.2, 14.6, and 12.9 N/mm², respectively which indicates a slight improvement in the mechanical properties of PVC composites (Figure 6). Meanwhile, the PVC/calcined kaolin composites have a remarkable higher elongation at break values than the corresponding PVC/raw kaolin composites which

Table 5: Calculations of density values of kaolin and calcined kaolin using density test results.

Sample Name	M_{total} (g)	M_{standard} (g)	M_{filler} (g)	ρ_{total} (g/cm ³)	ρ_{standard} (g/cm ³)	V_{total} (cm ³)	V_{standard} (cm ³)	V_{filler} (cm ³)	$\rho_{\text{filler}}^{**}$ (g/cm ³)	Average ρ_{filler} (g/cm ³)
Standard PVC	153	153	0	1.245	1.245	122.89	122.89	0	-	-
PVC/15 KL	168	153	15	1.306	1.245	128.64	122.89	5.75	2.61	2.59
PVC/30 KL	183	153	30	1.361	1.245	134.46	122.89	11.57	2.59	
PVC/45 KL	198	153	45	1.41	1.245	140.43	122.89	17.53	2.57	
PVC/15 CKL	168	153	15	1.307	1.245	128.54	122.89	5.65	2.66	2.63
PVC/30 CKL	183	153	30	1.363	1.245	134.26	122.89	11.37	2.64	
PVC/45 CKL	198	153	45	1.412	1.245	140.23	122.89	17.34	2.60	

*Mass of filler in grams

**Density of filler

**Figure 6:** The mechanical properties of PVC/raw kaolin composites (PVC/KL) and PVC/calcined kaolin composites (PVC/CKL) (a): Tensile strength; (b): Elongation at break.**Figure 7:** The thermal stability of PVC/raw kaolin composites (PVC/KL) and PVC/ calcined kaolin composites (PVC/CKL).

ensures the relative enhancement in the mechanical properties of PVC composites (Figure 6). These notable enhancements could be revealed as a result of the weakened interlayer Tetrahedral-Octahedral (T-O) bonds of the kaolin. Thereafter, the dispersion and interaction between PVC resins and calcined kaolinite layers were improved [32]. Good dispersion and interaction between

PVC resins and calcined kaolinite layers produce better mechanical properties of PVC composite [1].

The thermal properties of PVC composites The results of the thermal stability test indicate a general increase in the thermal stability of PVC/kaolin composites with increasing the raw kaolin content. (Table 4 and Figure 7)

The thermal stability was increased from 49 minutes in the PVC standard sample (without filler) to 54 minutes, 59 minutes and, 63 minutes in case of adding 15 phr, 30 phr, and 45 phr of raw Kaolin (KL), respectively. These results give an indication of the heat stabilizing effect of raw kaolin on PVC composites, which could be revealed because of the reasonable content of metal oxides in the raw kaolin. Metal oxides of calcium, iron and titanium enhance the thermal stability of PVC composites [1,3]. On the other hand, the thermal stability graph of PVC/calcined kaolin composites shows an initial decrease in the thermal stability values from 49 minutes for standard PVC composite sample to 34 minutes for PVC/15 phr calcined kaolin (Figure 7). It increases to 45 minutes and 52 minutes for 15 phr, 30 phr, and 45 of PVC/calcined kaolin (PVC/CKL) respectively. Thus, the thermal stability results of the PVC/kaolin composite are higher than its corresponding PVC/calcined kaolin composite sample. Reported that the pH of kaolin decreases directly with the gradual increase of the thermal treatment of kaolin till 700 °C and consequently, the acidity of kaolin increases [31].

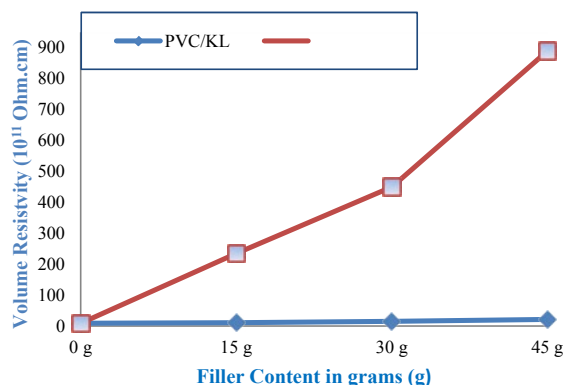


Figure 8: The Volume resistivity of PVC/raw kaolin composites (PVC/KL) and PVC/ calcined kaolin composites (PVC/CKL).

Therefore, the acidic character of the calcined kaolin decreases the efficiency of Ca/Zn stabilizer [33]. This criterion in addition to the decrease of iron oxides content by magnetic separation method are, most probably, the reasons for the differences between the thermal stability data of PVC/kaolin composite and their corresponding PVC/calcined kaolin composite. Moreover, the relative increase in thermal stability of 30 and 45 PVC/calcined kaolin than 15 PVC/calcined kaolin indicates that the calcined kaolin has heat stabilizing properties, which directly proportional to the increment of its content.

The electrical properties of PVC composites Volume resistivity data show a moderate enhancement in PVC/kaolin results with the gradual addition of raw kaolin content until valued $2.1469 \times 10^{12} \Omega\text{cm}$ for 45 PVC/kaolin composite (Table 4 and Figure 8).

However, these enhancements are too low to be compared with the effect of adding 15 phr of calcined kaolin in PVC composite. The volume resistivity of 15 PVC/calcined kaolin was estimated to be $2.35 \times 10^{13} \Omega\text{cm}$. The electrical volume resistivity of PVC composites increases particularly with increasing the calcined kaolin content to a value of $8.89 \times 10^{13} \Omega\text{cm}$ for 45 PVC/calcined kaolin composite (Figure 8). The high-volume resistivity values of PVC/calcined kaolin composites can be best explained as a result of the decrease of the low-resistance impurities such as; iron oxides, titanium oxides, and water content. This consequently increases the electrical resistivity of the resulted PVC/calcined kaolin compound [34-35].

CONCLUSIONS

In this study, raw kaolin from the Sinai Peninsula in north eastern Egypt was treated by the magnetic separation method and thermal calcination at 700°C process to prepare a calcined kaolin. Both of kaolin and calcined kaolin were used as fillers in the formulation of PVC composites. Chemical composition results by XRF analysis show that the $\text{TiO}_2\%$, Fe_2O_3 , and loss of ignition were decreased during the preparation of the calcined kaolin. The density of kaolin and calcined kaolin were calculated as about 2.59 g/cm^3 and 2.63 g/cm^3 , respectively. Thus, the density of kaolin was slightly Increased after physical and thermal treatments. The thermal stability of PVC/kaolin composites show a direct increase with adding raw kaolin, thus raw kaolin has a secondary stabilizing effect in addition to its filler character. Additionally, both of mechanical

and electrical properties of PVC composite improved significantly after using the calcined kaolin as a filler rather than the raw kaolin. The volume resistivity highlights a remarkable enhancement with increasing the content of the calcined kaolin. The present work adds another application of the kaolin ore deposit as a filler for PVC compounds, which is a vital material for everyday life usage. We do highly recommend its usage instead of other imported material at a high cost.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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