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Dielectric Properties of Potassium Hydroxide-Treated Palm Kernel Shell for Microwave-Assisted Adsorbent Preparation

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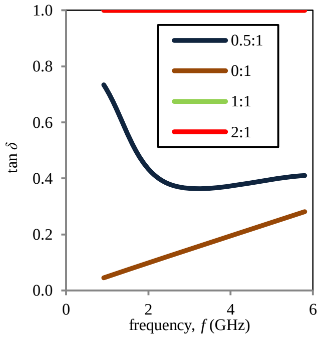
Graphical abstract



Abstract

This study was aimed to evaluate the dielectric properties of the reactants mixture at varying temperatures and concentrations for microwave-assisted adsorbent preparation, and to study the adsorptive characteristics of the resultant adsorbents. Palm kernel shell (PKS) was used as the precursor while potassium hydroxide (KOH) was employed as the activator. Different concentrations of KOH solution and ratios of KOH:PKS of 0:1, 0.5:1, 1:1, and 2:1 were prepared, and the measurement of the dielectric properties was performed using open-ended coaxial probe method. Results indicate that the relative dielectric constant, ε_r' is inversely proportional to the frequency, *f*. The trends of relaxation time, τ and penetration depth, D_P with respect to the frequency and temperature were also reported. Adsorbent prepared using a higher ratio of KOH shows a greater removal of methylene blue. Dielectric properties are imperative especially for selecting suitable operating conditions and in designing the applicator for effective preparation of adsorbent.

Keywords: Adsorbent; dielectric properties; palm kernel shell; penetration depth; potassium hydroxide; relaxation time

■1.0 INTRODUCTION

Carbonaceous agro-waste products such as coconut husk, palm kernel shell and empty fruit bunch are among the promising candidates of activated carbon–a material that is widely used in the removal of pollutants from water. The aforementioned precursors are chemically stable, possess high mechanical strength, and abundantly available at almost no cost. The use of these materials in waste water treatment may also help to reduce the disposal problems they formerly introduce [1].

Activated carbon can be prepared through chemical activation or physical activation where suitable activation temperature is usually attained by heat supplied by electric furnace [2-4]. Hot gases such as superheated steam and carbon dioxide (CO_2) are commonly used in physical activation. The raw material is first subjected to a carbonization process under an inert atmosphere followed by the activation of the resulting char in the presence of hot gases [3]. In the chemical activation, activators such as zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), potassium hydroxide (KOH), and potassium carbonate (K_2CO_3) are used in the single-step activation [4]. It requires the raw material to be impregnated with activator and then followed by thermal activation to create the pore structure. Chemical activation is preferred over physical activation due to the lower activation temperature, higher yield and shorter time for activation [3-5].

Recently, the use of microwave to assist the preparation of activated carbon has been widely reported in the literature [6-8]. Activated carbons with similar textural properties as that prepared through conventional heating could be obtained. Microwave offers some advantages over conventional heating, such as energy efficient process, shorter activation period and higher yield [9]. However, the dielectric properties of the reactant mixtures for the preparation of adsorbent are not extensively studied and poorly documented.

The behaviour of the material in the electromagnetic field at different frequencies and temperatures could provide a basis for the design of a large scale microwave applicator or continuous process microwave reactor that is economical and efficient for adsorbent preparation. The determination of dielectric properties is also important to understand the heating profiles of materials in a microwave heating process. The domestic microwave frequency is 2.45 GHz, and it should be noted that the dielectric properties of a material are not only depend on the frequency but also vary with moisture content, temperature and composition of the material [9].

The present work was aimed to investigate and quantify the related and important properties in microwave heating of potassium hydroxide-palm kernel shell mixtures and the resultant adsorbents at varying frequencies and temperatures. These include dielectric constant, ε_r', dielectric loss (or loss factor), ε_r'', loss tangent, tan δ, relaxation time, τ, and penetration depth, D_p. The resultant adsorbents were also tested to remove methylene blue dye solution. The trends and behaviour of the materials at the said conditions were reported and discussed.

■2.0 MATERIALS AND METHODS

2.1 Materials

Empty Palm kernel shell was obtained from Felda Taib Andak palm oil mill, located at Kulai, Johor. Potassium hydroxide (KOH) was obtained from R&M Chemicals, and was used without further purification.

2.2 Preparation of Adsorbent

Palm kernel shell (PKS) was ground to a uniform size of 2.5 ± 0.5 mm. One gram of PKS was mixed with potassium hydroxide (KOH) at weight ratios (KOH:PKS) of 0:1, 0.5:1, 1:1 and 2:1 in 50 mL distilled water. The mixture was transferred to the teflon mould, and the activation was performed in a 2.45 GHz microwave oven at 70% power intensity until the solid is completely dried. The resultant adsorbents were thoroughly washed with distilled water to a constant pH to remove the remaining salt and impurities. Then, the adsorbent materials were dried in an oven prior to use.

2.3 Dielectric Properties Measurement

The dielectric properties of the reactant mixtures and the resultant adsorbents were determined at varying operating frequency, f and temperatures, T. The dielectric properties were measured from 1 to 6 GHz using Keysight 85070E dielectric probe (Formerly Agilent Technologies) attached to the Keysight E5071C network analyzer. The samples was heated by a hot plate to vary the temperature from 25^{o}C to 45^{o}C. The dielectric probe aperture was immersed into the liquid samples or pressed to the solid samples prior to measuring the dielectric properties. The measurement was repeated for several times to obtain the average values.

2.4 Adsorptive Characteristics

The surface functional groups were determined by KBr disc method using Fourier Transform Infrared (FTIR) spectroscopy (Spectrum One, Perkin Elmer).

The removal of methylene blue dye was performed by adding 50 mg adsorbents to the conical flasks containing 50 mL methylene blue solution of varying concentrations (3-189 mg/L). The mixtures were allowed to equilibrate on orbital shaker at room temperature and 120 rpm for 72 h. The supernatant was removed and checked for concentration using visible spectrophotometer (Halo Vis-10, Dynamica) at wavelength of 608 nm. Blank sample was also prepared to represent initial concentration. The adsorption was determined as, $q_e = (C_o - C_e)$ (V/m), where q_e is the adsorption capacity (mg/g), C_o and C_e are respectively the initial and final equilibrium concentrations (mg/L), V is the volume of dye solution (L) and m is the mass of adsorbent (g).

■3.0 RESULTS AND DISCUSSION

3.1 Dielectric Properties of Materials

3.1.1 Effect of Salt Concentration

The trends of the relative dielectric constant, ε_r', loss factor, ε_r'' and loss tangent, tan δ for mixtures of palm kernel shell in potassium hydroxide solution from 1 to 6 GHz at 25°C are shown in Figure 1.

The dielectric constant, ε_r', gives the information on the ability of material to store the external electric field through polarisation mechanism. From Figure 1(a), it can be observed that the values of ε_r' decreased with increasing operating frequency, f. Generally, the ability of the material to store the microwaves energy upon the interaction with microwaves decreases because the values of ε_r' become low as the frequency increases. The high value of ε_r' at low frequency reflects the effect of space charge polarization and/or conducting ionic motion, whereas the decreases of ε_r' with increasing of frequency, f may be due to the decrease in space charge carriers or interfacial polarization [10-11].

Increasing the salt concentration from weight ratio 0:1 to 0.5:1 somewhat decreases the dielectric constant, ε_r' along the frequencies studied. Adding the salt or electrolyte into the mixture increases the conductivity of the solution, at the same time decreasing the polarization of the molecules in the mixture. The profiles for the salt mixtures with ratios 1:1 and 2:1 are not shown in Figure 1(a) due to solution becomes too saturated and behaves like a lossy conducting material (tan δ=1.0) for all frequencies with very high values in ε_r' and ε_r''. The high values of ε_r' in the conducting dielectric materials may be related to the accumulation of charges that leads to the polarization space charge [10-11].

The profiles of ε_r'' and tan δ for ratios 0.5:1 and 1:1 in Figure 1(b) and (c) are similar. It suggests that tan δ of the materials depends largely on the dielectric loss, ε_r''.

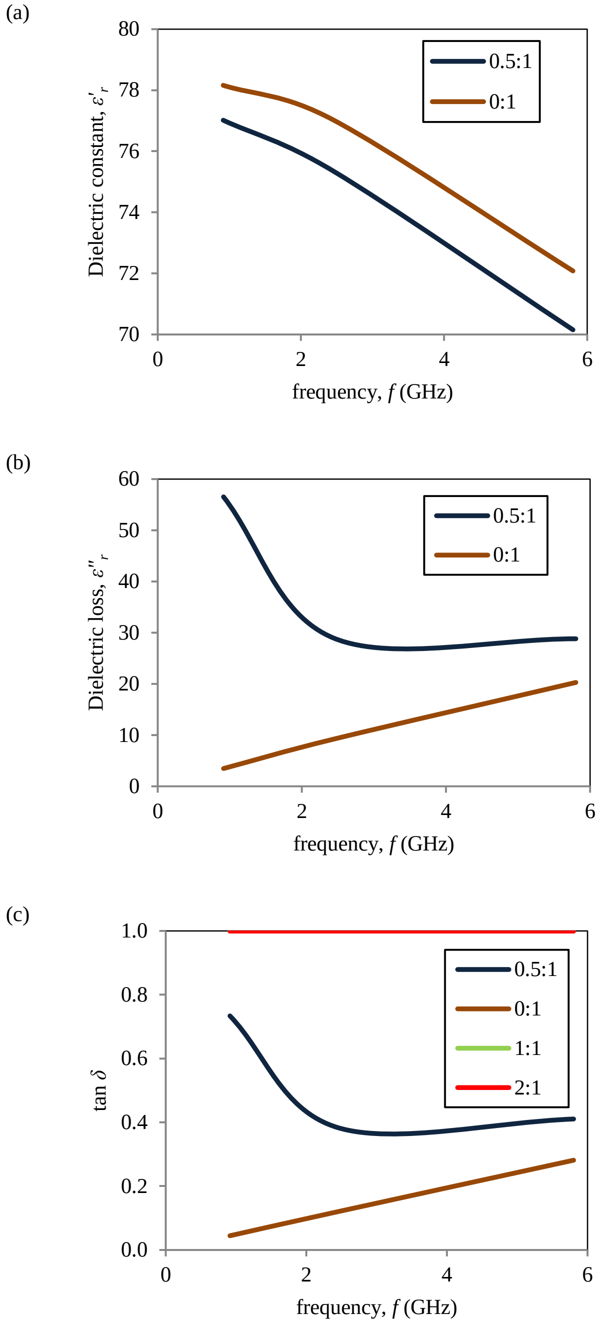


Figure 1 Effect of frequency on dielectric properties of potassium hydroxide-palm kernel shell mixtures at 25°C: (a) dielectric constant, (b) dielectric loss and (c) tan δ

By definition, tan δ describes the efficiency of a material to dissipate the store energy (microwaves) as heat. Both ε_r'' and tan δ for palm kernel shell particles in water increased with increasing operating frequency. It suggests that the power absorption and heating rate of the mixture in microwave is better and faster at higher frequency. On the contrary, the mixture with weight ratio 0.5:1 (palm kernel shell particles in 0.01 g/mL KOH) showed a decreasing tan δ with the increasing frequency from 0.915 to 2.45 GHz, then experiencing a slight increase as the frequency increases to 5.8G Hz. This might be due to the change in the electrical conductivity, σ of the mixture. It also implies that the microwave absorption of electrolyte mixture is more efficient at lower frequency.

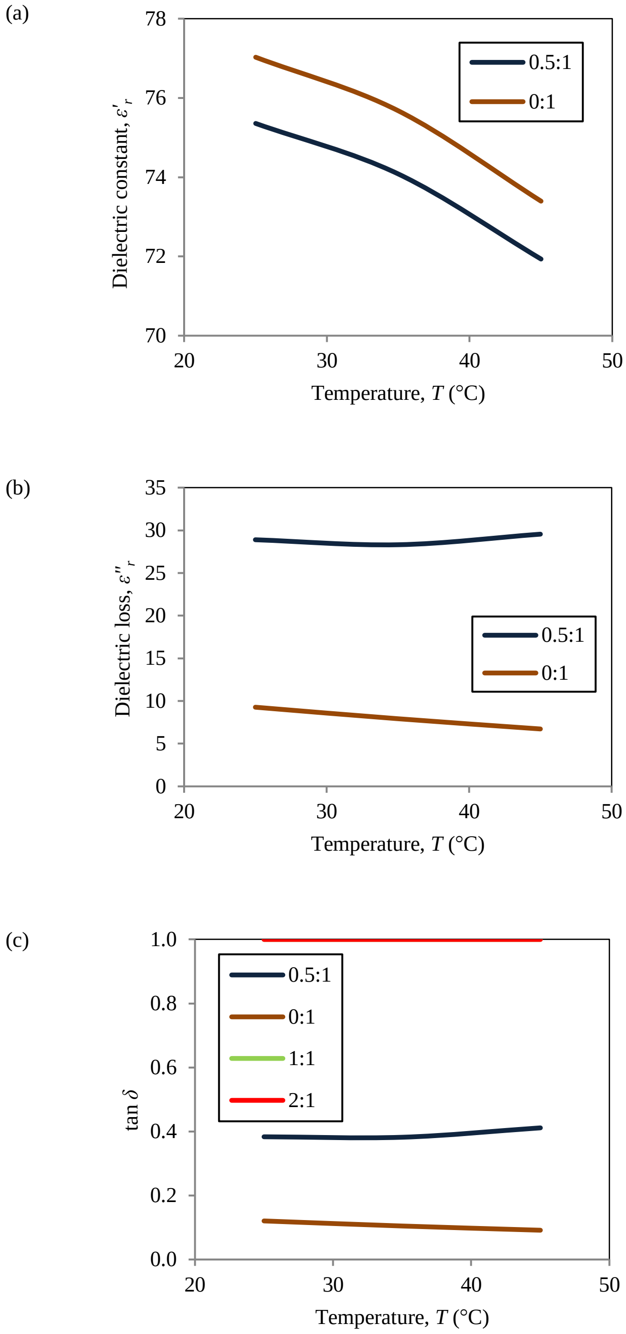


Figure 2 Effect of temperature on dielectric properties of potassium hydroxide-palm kernel shell mixtures at 25°C: (a) dielectric constant, (b) dielectric loss and (c) tan δ

At all frequencies studied, a slightly concentrated KOH salt solution (ratio 0.5:1) exhibits a better ability to be heated using microwave, and better material characteristics to absorb and dissipate microwave energy as heat compared to pure water (ratio 0:1). Because both ratios 1:1 and 2:1 are saturated, the tan δ for these materials are equal to unity.

A few attempts were also made to determine the dielectric properties, ε_r' of the liquid without the presence of solid particles (figures not shown). The trends of dielectric properties for with (Figure 1) and without palm kernel shell particles are identical, suggesting that the dielectric properties are highly dependent on the concentration of the electrolyte.

3.1.2 Effect of Solution Temperature

The dielectric properties of the materials were respectively investigated at 25, 35 and 45°C, at fixed frequency of 2.45 GHz. The results are presented in Figure 2. From Figure 2(a), it was found that the values of ε_r' decreased with increasing temperature from 25°C to 45°C. Similar trends in values of ε_r'' and tan δ were observed for materials with ratios 0:1. This trend may be related to the viscosity and relaxation time of the material change with the temperature. The trend is similar for most solvents used in chemical synthesis such as water, ethanol, methanol and acetone [12-13]. On other hand for ratio 0.5:1, the values of ε_r'' and tan δ are slightly increased with temperature. It shows that the propensity of the heated material to maintain its ability the absorb microwave energy is more stable with the presence of low concentration of salt or electrolyte. This result also suggests that the temperature and electrolyte concentration dependence of palm kernel shell's dielectric properties at the fixed frequency.

At relatively low temperature, the charge carriers (electrolyte) slowly orient themselves with respect to the direction of applied field. As a result, the material exhibits weak contribution to the polarization and the dielectric behaviour. While, at relatively high temperature, the bond charge carriers get enough excitation thermal energy to be able to obey the change in the external field more easily. Space charge contribution to the polarization may also be attributed to the purity of the material. This in return enhances their contribution to the polarization leading to an increase of dielectric behaviour at higher temperature.

3.1.3 Effect of Solid Temperature

Table 1 displays the comparison of the dielectric constant, ε_r' and loss tangent, tan δ of solid adsorbents at 25 and 45°C.

Table 1 Comparison of ε' and tan δ of solid materials (resultant adsorbents) at different frequencies and temperatures

Sample	25°C					
	0.915GHz		2.45GHz		5.8GHz	
	ε'	tan δ	ε'	tan δ	ε'	tan δ
PKS	2.19	0.0293	2.10	0.0510	2.24	0.0509
Ratio 0:1	2.05	0.0243	1.99	0.0406	2.10	0.0428
Ratio 1:1	2.11	0.0194	1.97	0.0393	2.09	0.0398
Ratio 2:1	2.37	0.0152	2.16	0.0359	2.24	0.0429
Sample	45°C					
	0.915GHz		2.45GHz		5.8GHz	
	ε'	tan δ	ε'	tan δ	ε'	tan δ
PKS	2.32	0.0217	2.19	0.0392	2.33	0.0484
Ratio 0:1	1.97	0.0191	1.84	0.0432	1.97	0.0392
Ratio 1:1	2.14	0.0225	1.98	0.0413	2.13	0.0423
Ratio 2:1	1.90	0.0216	1.90	0.0403	2.00	0.0363

In general, solid materials demonstrate lower values of ε_r' and tan δ than their liquid counterparts. It gives a clear indication that water and low concentration electrolytes are better loss materials for microwave. However, the variation in the dielectric properties of the solid materials could shed some light on the design aspects in microwave-assisted adsorbent preparation.

From Table 1, the tan δ of solid materials increased with the increasing frequency. This is true for the two temperatures studied. It is suggested that the microwave heating for solid material could be effectively done at a moderate frequency (e.g., 2.45 GHz). It is also obvious that the tan δ for adsorbent materials (ratios 1:1 and 2:1) increased at 45°C as opposed to the non-impregnated samples (PKS and ratio 0:1). This could be due to the increase of carbon content and aromatic structure within the material as a result of the chemical (KOH) activation, which in turn improves the microwave heating process [9].

3.1.4 Relaxation Time and Penetration Depth

Relaxation time, τ is defined as the time taken by molecules to realign themselves to their original position when the electromagnetic field is removed. The equation for the relaxation time is given as [14],

$$\varepsilon_r' = -(\omega\varepsilon_r'')\tau + \varepsilon_s \qquad (1)$$

where, ω is angular frequency (ω=2πf), and ε_s is static permittivity when ω=0. The relaxation time, τ was obtained from the slopes of ε_r' against -$\omega\varepsilon_r''$. Penetration depth, D_p is the depth (in cm) that the microwave power can penetrate in the material before its surface value has fallen to 1/e (0.368) [15-16]. The penetration depth is given as,

$$D_p = \frac{\lambda}{2\pi\varepsilon_r''}\sqrt{\varepsilon_r'} \qquad (2)$$

where, λ (= c/f) is the operating wavelength and c (= 3×10^8 m/s) is the velocity of light in free space. The relaxation time, τ and penetration depth, D_p of the studied materials at 25 and 45°C are summarized in Table 2.

In general, the relaxation time, τ decreased with increasing operating frequency, f at the two temperatures studied. This could be explained as the electromagnetic energy is stronger at high operating frequency, f and could obstruct the dipoles or molecules to align themselves, which thereafter leads to partial polarization and inefficient heating [17]. The penetration depth, D_p is a very important factor in order to know the ability to heat the material efficiently throughout its interior. Likewise, the penetration depth, D_p also decreased as the operating frequency, f increased from 0.915 to 5.8 GHz. If the frequencies are higher, the electromagnetic energy might get absorbed on the surface of the materials, and will penetrate only a short distance. Thus, the higher the penetration depth, D_p the more efficient the material can be heated. Adsorbent (ratio 2:1) exhibits higher D_p value that is about twice as much as that of raw palm kernel shell at the frequency of 0.915 GHz, while 0.01g/ mL KOH solution shows a drastic decrease in D_p compared to water at the same frequency. The former is possibly due to the enrich graphitic structure within the material matrix, while the latter could be a result of the increasing conductivity in the solution. As the temperature increases to 45°C, τ was found to decrease, while D_p remains slightly unchanged. The values are fairly stabilized at 2.45 GHz.

It is obvious that the frequency plays a vital role in heating the materials under microwave. As the dielectric properties of a material are unique, it is therefore imperative to select suitable frequency for effective heating via microwave.

Table 2 Relaxation time and penetration depth of materials at different frequencies and temperatures

Material	Frequency (GHz)	25°C		
		τ (ps)	D_p (cm)	ε_s
Water	0.915	9.93	13.6	78.2
	2.45	8.53	1.87	
	5.8	7.99	0.346	
0.01g/mL KOH	0.915	22.1	0.544	76.0
	2.45	10.4	0.437	
	5.8	7.78	0.214	
PKS	0.915	202	120	2.19
	2.45	164	26.4	
	5.8	149	10.8	
Ratio 2:1	0.915	1168	223	2.37
	2.45	105	36.9	
	5.8	105	12.8	
Material	Frequency (GHz)	45°C		
		τ (ps)	D_p (cm)	ε_s
Water	0.915	4.14	17.8	74.5
	2.45	8.03	2.51	
	5.8	7.55	0.463	
0.01g/mL KOH	0.915	15.0	0.464	72.4
	2.45	6.79	0.399	
	5.8	6.56	0.231	
PKS	0.915	32.0	158	2.32
	2.45	23.9	33.6	
	5.8	128	11.2	
Ratio 2:1	0.915	344	175	1.90
	2.45	74.7	35.1	
	5.8	237	16.1	

3.2 Adsorptive Characteristics

The yield of solid materials (adsorbents) are in the order of PKS (100%) > ratio 0:1 (88.3%) > ratio 1:1 (66.7%) > ratio 2:1 (58.8%). Increasing the impregnation ratio usually decreases the yield; the weight losses are due to the release of volatile products as a result of intensifying dehydration, elimination reactions and gasification of surface carbon atoms by potassium hydroxide (dehydrating agent) [4]. The pH values of the solid materials were recorded as 5.7, 7.8, 7.9 and 9.3 for PKS, and ratios 0:1, 1:1 and 2:1, respectively.

Figure 3 displays the FTIR spectra for palm kernel shell and the adsorbent derivatives. The microwave dried-palm kernel shell (ratio 0:1) displays a similar FTIR profile as the raw palm kernel shell. It signifies that both materials possess identical surface functional groups.

Peaks centred at 3300 cm^{-1} generally indicate the presence of physisorbed water and –OH stretching band. The small intensity peaks located at 600 to 1400 cm^{-1} could be assigned to alkanes, alkenes and aromatic structures. The C=O stretching vibrations of simple ketones and carboxylic acids occur at frequency around 1710 cm^{-1}. From Figure 3, it is obvious that the KOH-treated palm kernel shell adsorbents (ratios 1:1 and 2:1) show spectra with decreasing intensity compared to their precursor (PKS). Potassium hydroxide is a dehydrating agent, and its presence in the microwave-assisted adsorbent preparation could accelerate the removal of the surface functional groups [18-19].

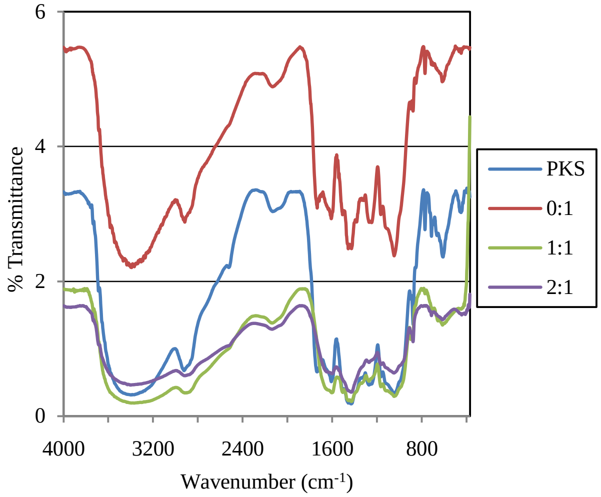


Figure 3 FTIR spectra of palm kernel shell and the adsorbent derivatives

Figure 4 shows the adsorptive ability of solid materials in methylene blue dye removal. At initial dye concentration of 3.1 mg/L, all solid material show identical removal performance at 2.5 mg/g. At initial concentration of 20 mg/L, raw PKS and the microwave-dried one demonstrate only a slight increase in the removal of methylene blue. So, further methylene blue uptake at greater initial concentrations was not performed for these two adsorbents (Figure 4). As the initial concentration increases to 189 mg/L, the KOH-treated adsorbents show a tremendous increase in the uptake of methylene blue. The highest removal of about 130 mg/g was recorded within the concentration range studied.

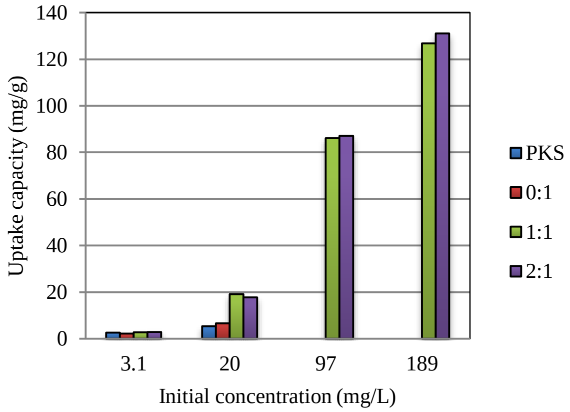


Figure 4 Removal of methylene blue by palm kernel shell and the adsorbent derivatives

The removal of methylene blue is probably driven by the basic surface of the adsorbent. As already mentioned, the pH of the treated adsorbents increased upon the treatment. The opposite charges between the surface and the cationic methylene blue molecules could be the mechanisms for the improved adsorptive ability of the treated adsorbents [20-21]. The pH of material can also affect the chemistry in the solution and the dissociation of functional groups on the active sites of the adsorbent. This subsequently leads to a shift in the rate and equilibrium characteristics of the adsorption process [4, 20].

■4.0 CONCLUSION

The dielectric properties are dependent on frequency, concentration, phase and temperature. The presence of small concentration of salt or electrolyte improves the ability of the material to be heated using microwave energy. The material is able to absorb more microwave energy in the presence of low concentration of KOH solution. However, the penetration depth of the electrolyte solution decreased with increasing temperature. Also the relaxation time, τ and penetration depth, D_p decrease with increasing of frequency. Therefore, suitable operating frequency, f preferably at the moderate regime (2.45 GHz) should be selected for effective heating by microwave, by which a higher penetration depth, D_p would help to heat the material more efficiently. The potassium hydroxide-treated adsorbents demonstrate a superior removal of methylene blue dye from water.

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