Experimental Investigation on The Influence of Temperature on The Adsorption of Henna Extract on Kaolinite in Reducing Surfactant Adsorption

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ABSTRACT: The adsorption ability of henna extract as anenvironment-friendly and easily available sacrificial agent was investigated. Fourier transform infrared-attenuated total reflectance (FTIR-ATR) was used to characterized henna extract and kaolinite. The adsorption of henna extract on kaolinite was done using Ultraviolet-visible spectroscopy (UV-Vis). The influence of temperature on the henna extract adsorption on kaolinite was studied. The mechanisms of the adsorption process were interpreted. Also, henna extract performances in reducing the adsorption of surfactant were assessed. The outcome shows that henna extract adsorption on kaolinite was decreased with increasing temperature. The adsorption value decreased from 7.88 to 7.04 mg/g from the temperature of 25°C to 75°C due to the increased of the kinetic energies of the henna extract molecules. A reduction of 38% of surfactant adsorption was observed and showed a profound drop in the adsorption of surfactant in the presence of henna extract suggesting a possibility to be utilized as a sacrificial agent in reducing the adsorption of surfactant.
Keywords: Henna extract, Adsorption, Kaolinite, Temperature.

I. INTRODUCTION

Surfactant flooding has performed a vital part in enhanced oil recovery (EOR) by being utilized to reduce the interfacial tension (IFT) of oil and water to improve the displacement efficiency through oil recovery [1]. However, the surfactant adsorption on reservoir rock may impact the deprivation of the concentration of surfactant, which may yield them less competent and unproductive [2].

Due to the adsorption phenomenon of surfactants, endeavors have been made to lessen the adsorption of surfactant on reservoir rocks. A few investigations have been done on the usage of similarly charged surfactant on the same surface charge of the rock and concluded that anionic surfactant adsorption is decreased on sandstone, and the adsorption of cationic surfactant is decreased on carbonate owing to the repulsion of electrostatic between the adsorbent and the type of surfactants used [3,4].

Applications of sacrificial agent (SA) are thought to be a promising method in reducing surfactant adsorption. The SA is a material that is injected in a way to significantly inhibit or conceal all probable adsorption sites of the rock within the hydrocarbon formation. Weston et al. [5] have found that the capability of the molecules of surfactant in forming admicelles on the solid surface is the primary reason adsorption occurs. The principle reason SA strategy is exceptionally engaging for is to keep the development of these admicelles.

In this research, henna, a natural plant-based was investigated to be a potential SA. *LawsoniainermisL.* known as henna has been applied as a corrosion inhibitor [6,7] and work done by Moslemizadeh et al. [8] revealed that henna extract managed to reduce the swelling of sodium bentonite more compared to exposing sodium bentonite to polyamine and potassium chloride due to its inhibitive capability. Phenolic compounds that present in the henna leaves are the core constituents that contributed to the popularity of henna in specific fields as mentioned above [9,10]. Fig. 1 shows the molecular structure of one of the phenolic compounds that are in the henna leaves which is gallic acid [10].
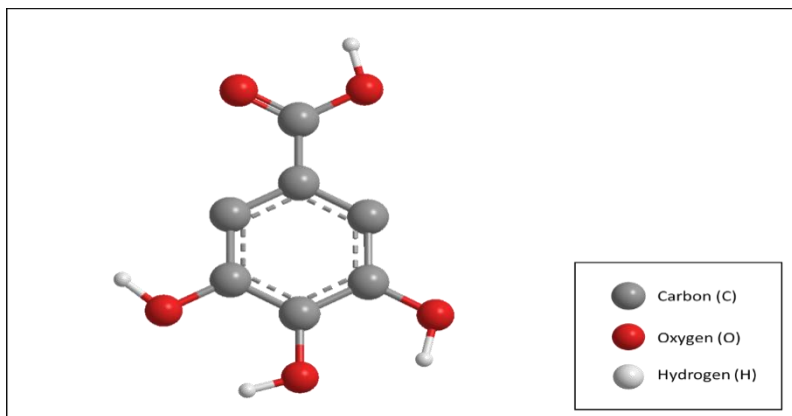
Fig. 1. Molecular structure of gallic acid.

The adsorption of surfactant at various temperatures have been investigated by some researchers. Temperature significantly influences the adsorption of surfactant on reservoir rock surface as specified by Beraet al. [11]. They came to the conclusion that with an increase in temperature, the adsorption capacity decreases. These observations are consistent with the one investigated by Yekeenet al.[12] and Khan & Zareen[13]who uncovers the trend of declining of adsorption with respect to the increase of temperatures. Be that as it may, the influence of temperature on the adsorption of henna extract on kaolinite is still unclear. Furthermore, there is an absence of detailed knowledge of the effect of temperature on the ability of henna extract as an SA in reducing surfactant adsorption.

The inspiration driving this work is to grasp a comprehension into the adsorption behavior of henna extract on kaolinite and its ability in reducing surfactant adsorption with the influence of temperature. The mechanisms of the adsorption process were analyzed. This study expects to validate to the point that henna extract could be used as an SA in minimalizing the surfactant adsorption.

II. MATERIALS AND METHODS

2.1. Materials

Fresh henna leaves were gathered from henna trees in Johor, Malaysia. Methanol of 99.9% (Acros Organics (USA)) was used as the solvent in methanolic extraction. Anionic surfactant, sodium dodecyl sulfate (SDS) of 98% purity with a molecular weight of 288.38 g/mol manufactured by Fisher Chemical (UK) was used for the surfactant adsorption. Kaolinite was obtained from Fisher Chemical (UK). Deionized water (DIW) was used for all experiments. All substances utilized were of scientific quality and were used as acquired devoid of added purifications.

2.2. Preparation of Henna Powder

The fresh leaves of henna were dried at room temperature and then grounded into powder using an electric blender. The henna powder was carefully packed in an airtight BPA free container and stored under room temperature until further used.

2.3. Characterization of Henna Extract and Kaolinite

2.3.1. FTIR-ATR Analysis

The FTIR-ATR was conducted using Perkin Elmer FTIR Spectrometer (USA). Functional groups of henna extract and kaolinite were identified by the spectrometer by observing the vibrational motion of bonds in the molecules. The spectra were measured in the range of 650-4000 cm^{-1} with a scan resolution of 2 cm^{-1}. The FTIR data were documented in the transmittance mode. The pattern of the spectrum was examined and compared to the IR absorption table to determine the functional groups encompassed in the samples.

2.3.2. XRD Analysis

The kaolinite sample was further characterized by using the X-ray diffraction (XRD) in the continuous scanning mode on SmartLab X-Ray Diffractometer (Rigaku, Japan) operated at 40 kV and a current of 30 mA with Cu-K$_\beta$ filter and Cu-K$_\alpha$ radiation source (λ = 0.154056 nm). Particle size ought to be fine to attain a tolerablestatistical representation of the components and their numerous diffracting crystal planes and to evade diffraction-related artifacts [14]. All the patterns were collected at room temperature with steps of 0.02^o in the 2θ range of 3°-100°. The measurements were taken at room temperature with a scan rate of 8.2551^o per minute.

2.4. Preparation of Henna Extract and Surfactant Solutions

Solutions of henna extract were prepared in standard 250 ml Erlenmeyer flasks. Henna extract was weighed and transferred into the flask, then DIW water was added to the required volume. Henna extract concentrations were prepared in the range of 3000 to 8000 mg/L. Surfactant solutions were preparedsimilarly as henna extract solutions. Different concentrations of surfactant were preparedin the range of 1000 to 5000 mg/L.

2.5. Adsorption Experiments

The adsorption of henna extract on kaolinite was determined using depletion method, that was, the differences between the concentrations of henna extract before and after adsorption on kaolinite. Kaolinite of 6 g was mixed with 30 mL of henna extract solutions. The mixture was then agitated in a temperature controller shaker using IKA KS 3000 I control (USA)at 180 rpm for 24 hours at 25°C under atmospheric pressure to reach equilibrium. They were then centrifuged using Rotofix 32A (Hettich Zentrifugen, Germany) at 4000 rpm for 30 min to isolate the buoyant liquid. The concentrations of henna extract in the buoyant liquid was measured with a UV-Vis spectrophotometer. Adsorption amount at equilibrium time, q_e (mg/g), was calculated using (Equation (1)). Same procedures were applied in calculating the adsorption of surfactant on kaolinite. Surfactant concentrations of 1000 mg/L to 5000 mg/L were chosen to determine the surfactant adsorption on kaolinite. In evaluating henna extract performances in reducing surfactant adsorption on kaolinite, the solution mixtures of henna extract and kaolinite were filtered out, leaving only kaolinite behind. Then, 30 mL of surfactant solution (concentration of 2000 mg/L) were mixed with the pretreated kaolinite with henna extract solution. The mixtures were then left to reach equilibrium for 24 hours throughout which they were recurrently shaken in a temperature controller shaker at 180 rpm. After equilibrium was reached, the mixtures were centrifuged, and the buoyant liquid was examined using UV-Vis spectrophotometer. Adsorption amount at equilibrium time, q_e (mg/g), was calculated using (Equation (1)).

$$q_e = (C_o - C_e) \times \frac{V}{m} \qquad (1)$$

where q_e is the adsorption of henna extract and surfactant on kaolinite (mg/g), C_o and C_e are the henna extract and surfactant concentrations before and after the adsorption experiment, respectively (mg/L), V is the volume of henna extract and surfactant solutions added to the volumetric flask (L) and m is the total mass of the kaolinite added (g).

III. RESULTS AND DISCUSSION

3.1. Characterization of Henna Extract and Kaolinite

3.1.1. FTIR Analysis

The FTIR-ATR spectrum of henna extract was analyzed and depicted in Fig. 2. A wide absorption band at 3311 cm^{-1} was seen. Those bands were appointed to the vibration of the hydroxyl groups. A similar observation was reported[6,15]. The vibration of the aliphatic C-H group appeared at 2926 cm^{-1} and 2855 cm^{-1}. The appearance of these functional groups is similar to the ones reported by other researchers [16]. The peaks at 1712 and 1632 cm^{-1} were assigned to the C=O bond. A similar view was noted by Safie et al.[17]. The peaks at 1510, 1449, 1400 and 1366 cm^{-1}were credited to the vibration of C=C in the aromatic benzene rings. Identical peaks were observed by several researchers [6,18]. The peaks at 1030 and 1064 cm^{-1} can be ascribed to the vibration of C-OH of the phenolic group as these peaks resembled the trend that was perceived by Saadaoui et al. [19]. A summary of the IR spectra of henna extracts is displayed in Table 1.
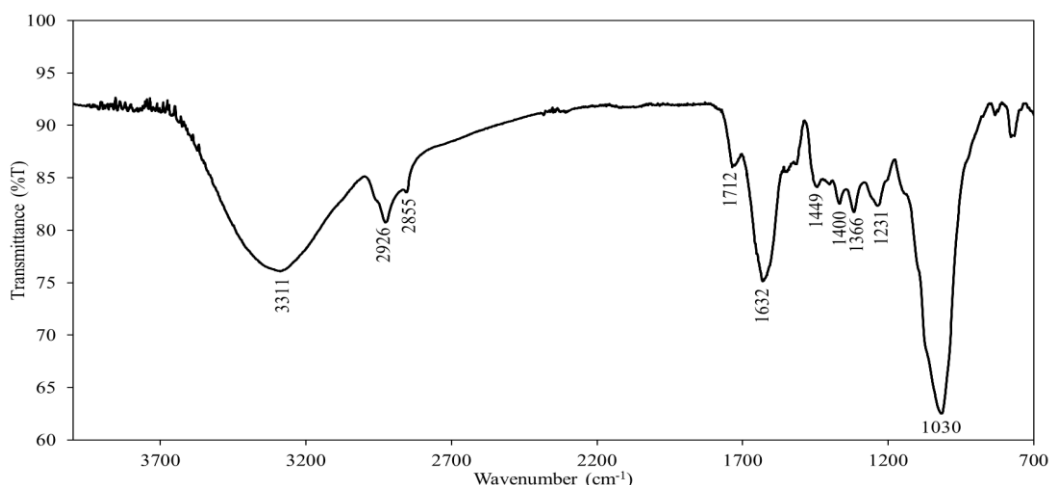
Fig. 2. FTIR-ATR spectrum of henna extract.

Table 1 Assignment of functional group frequency in the spectra of henna extract.

Wavenumber (cm^{-1})	Functional groups
3311	OH group
2926, 2855	Aliphatic CH group
1712, 1632	C=O group
1449, 1400, 1366, 1231	C=C group in aromatic benzene ring
1030	C-OH of phenolic group

Fig. 3 presents the infrared spectra of kaolinite. The FTIR-ATR spectrum of kaolinite shows bands at 3690, 3669, and 3651 cm^{-1} credited to the hydroxyl modes of the inner surface hydroxyl groups and the absorption at 3620 cm^{-1} was attributed to the hydroxyl modes of the inner hydroxyl groups as similar bands were conveyed by Balan et al. [20] and Li et al. [21]. Si-O and Si-O-Si stretching vibration were seen at 1113 cm^{-1}, 1023 cm^{-1}, and 1000 cm^{-1} as similar results were obtained by Dawodu and Akpomie [22]. The absorption band at 938 cm^{-1} tallies to the inner surface Al-OH group whereas the band at 913 cm^{-1} resembles the inner Al-OH group and the bands were close to the ones reported by Duarte-Silva et al. [23]. The bands that appeared at 792, 755, and 685 cm^{-1} were attributed to Si-O-Al and OH group. These groups are comparable to the ones determined by Ming and Spark [24] and Saikia and Parthasarathy [25]. A summary of the IR spectra of kaolinite is displayed in Table 2.
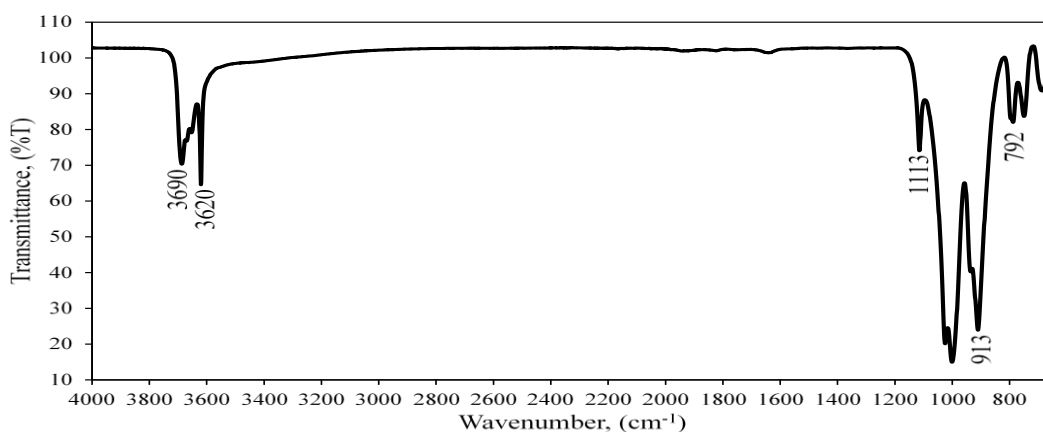

Fig. 3. FTIR-ATR spectrum of kaolinite.

Table 2 Assignment of functional group frequency in the spectra of kaolinite.

Wavenumber (cm^{-1})	Assignments
3690, 3669, 3651	Hydroxyl modes of inner surface hydroxyl groups
3620	Hydroxyl modes of inner hydroxyl groups
1113, 1023, 1000	Si-O and Si-O-Si groups
938	Inner surface Al-OH group
913	Inner Al-OH group
792, 755, 685	Si-O-Al and OH groups

3.1.2.   XRD Analysis

The XRD pattern of kaolinite is presented in Fig. 4. The diffraction peaks might be credited to kaolinite and agreed with the standard data given in the ICDD card, 01-078-1996. No impurities were seen. Furthermore, the intensities of the diffraction peaks were high, and the peaks were sharply specified that kaolinite was vastly crystalline and comprised wholly of kaolinite.
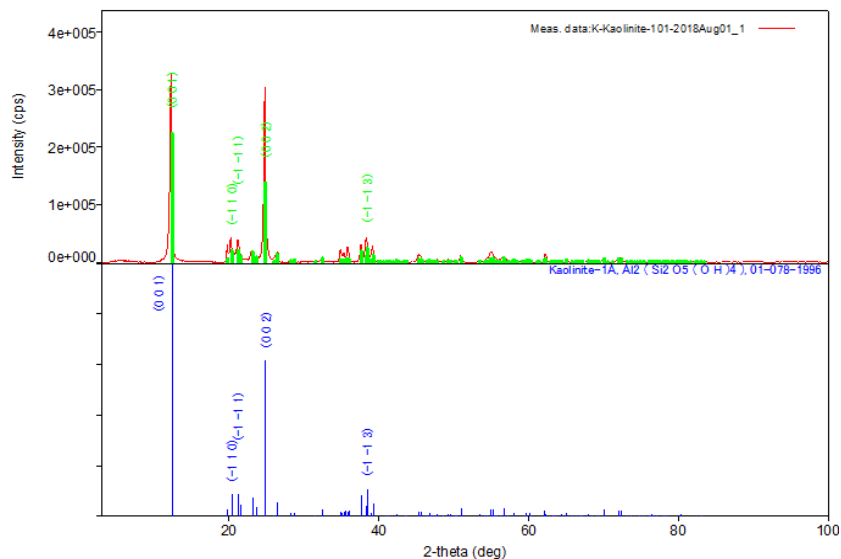
Fig. 4. XRD diffractogram of kaolinite.

3.2. Adsorption Experiments
3.2.1. Effect of Temperature on The Adsorption of Henna Extract on Kaolinite

The effect of temperature on henna extract adsorption was studied at 25, 45, 65 and 75°C. Fig. 5 depicted henna extract adsorption at various concentrations on kaolinite at different temperatures.
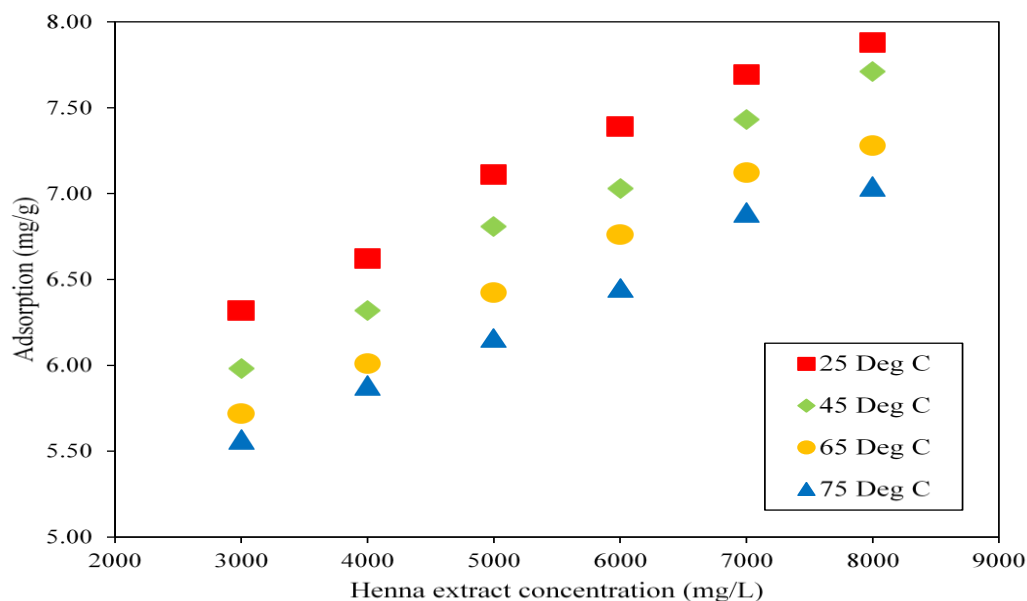

Fig. 5. Adsorption of henna extract on kaolinite at different temperature.

A decrease in henna extract adsorption on the adsorbent with an increase in temperature was observed. The adsorption of henna extract on kaolinite decreased from 7.88 to 7.04 mg/g with increasing temperature. Increase in temperature causes an increase in the mobility of henna extract molecules. Hence, the chance of physical contact between henna extract molecules and the kaolinite surface decreases [26]. Furthermore, when the temperature increased further, the kinetic energies of henna extract molecules became higher than the potential attractive forces of the active sites on the surface of kaolinite [27], leading to a decrease in the adsorption of henna extract on kaolinite, which may explain this behavior.

These results are in agreement with the literature that mentions that adsorption decreases when the temperature is increased. Chen and Lu [28] investigated the CO_2 adsorption capacity of kaolinite and found that the CO_2 adsorption capacity of kaolinite decreased with an increase in the adsorption temperature. Another study by Oliveira et al. [29] found that a rise in temperature involved a decreased in the adsorbed amount of diclofenac (micropollutant) on organoclays. A study by Paria and Khilar[30]observed that an increase in

temperature leads to a considerable decrease in the maximum quantity adsorption of surfactants. Overall, the reduction in adsorption at higher temperature is expected as an increase in the kinetic energy of the species which then resulted in a decrease of aggregation of the henna extract molecules on the surface of kaolinite [31].

3.2.2. Influence of Temperature on The Adsorption of Surfactant on Kaolinite without The Presence of Henna Extract

Surfactant adsorption in ambient temperature (25°C) and temperature of 45°C was executed,and the results were exhibited in Fig. 6. From the figure, the adsorption decreased when increasing the temperature from 25°C to 45°C.
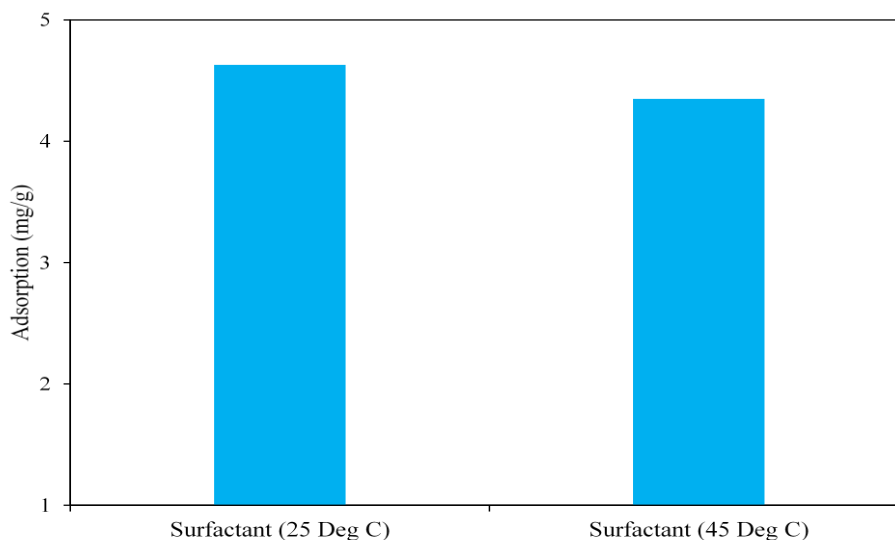

Fig. 6. Surfactant adsorption with the influence of temperature.

The reduced adsorption from 4.63 mg/g to 4.35 mg/g for kaolinite, is owing to the active molecules that have high kinetics energies which made the interactions between the kaolinite surface reduced. Some of the studies which investigated the effect of temperature in surfactant adsorption, agree with the results [32,33].

3.2.3. Influence of Temperature on The Adsorption of Surfactant on Kaolinite with The Presence of Henna Extract

Henna extract performance in reducing surfactant adsorption at different temperature was investigated. Based on results in section 3.2.2, the temperature of 25°C and 45°C gave the highest henna extract adsorption rather than 65°C and 75°C, thus these conditions were further evaluated in the surfactant adsorption.

It can be noted in Fig. 7, that the adsorption of surfactant was reduced in the presence of henna extract in ambient temperature. Henna extract managed to reduce surfactant adsorption on kaolinite from 4.63 mg/g to 2.88 mg/g. A reduction in surfactant adsorption with henna extract was calculated as 38%. Also, in the same figure, it displays the performance of henna extract at a temperature of 45°C which showed a similar declining trend when the temperature was at 25°C. Henna extract succeeded in decreasing the surfactant adsorption from 4.35 mg/g to 2.91 mg/g which corresponds to 33% of reduction.
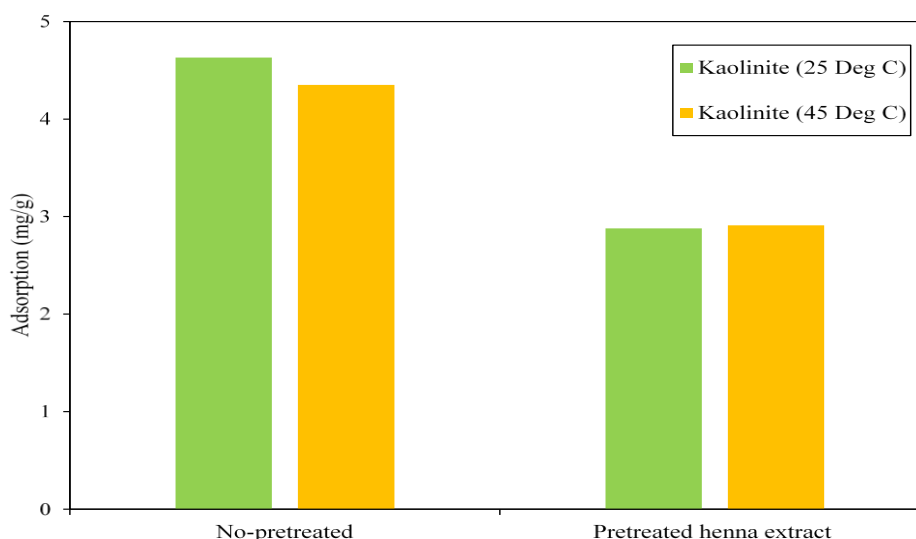
Fig. 7. Henna extract performances on the reduction of the surfactant adsorption with the influence of temperature.

Although henna extract was successful in reducing the surfactant adsorption at the temperature of 45°C, nevertheless, a declined in the percentage of surfactant adsorption with henna extract was observed. The adsorption decreased from 38% to 33% and can be due to the diminished binding strength of henna extract as discussed in section 3.2.2 above. As a consequence, surfactant molecules were capable of binding to the kaolinite surface and henna extract molecules via hydrogen bonding and hydrophobic interactions, thus, produced less surfactant molecules.

IV.   CONCLUSION

The existence of these functional groups in henna extract, hydroxyls, carbonyls, phenolics, aromatic benzene ring, and aliphatic carbon-hydrogen groups made the adsorption process conceivable. Kaolinite was characterized and used as an adsorbent to investigate the influence of temperature on henna extract adsorption on kaolinite and to evaluate the performances of henna extract as an SA in reducing surfactant adsorption. The adsorption of henna extract with several temperatures has been investigated. It was noticed that by increasing the temperature from 25°C to 75°C, the adsorption of henna extract was decreasing. This was because of the henna extract molecules gained higher kinetic energies when the temperature was raised, consequently, reduced the time spent on the surface of kaolinite. The experimental results at different temperatures established that by increasing the temperature, the adsorption of surfactant decreased. Similar findings were obtained with the henna extract adsorption. It was observed that the highest adsorption occurred was at room temperature rather than elevated ones. Higher temperatures tend to increase the kinetic energies of the molecules and hence, increase the entropy of the systems. This led to the shorter time taken for the molecules to make contact with the kaolinite surface, therefore, caused in the decrease of the molecule's aggregation on the surface of the adsorbent. When the temperature was at 25°C, a reduction of 38% of the surfactant adsorption on kaolinite can be perceived. This is due to the coating of the henna extract on the surface of the adsorbent, which led to the inability of the surfactant to adsorb on the surface of kaolinite. However, upon increasing the temperature to 45°C, a decrease of 33% in the surfactant adsorption was noticed. The rationality behind this was, by the increased kinetic energies of the molecules. Since some of the henna extract molecules could not afford to adsorb on the surface of kaolinite, there was a possibility that some of the surfactant molecules contentedly adsorbed on the surface leading to the increased quantity of surfactant adsorption.

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