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Potential of kaolinite as adsorbent to remove anionic surfactant from simulated industrial wastewater

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ABSTRACT

This paper describes the removal of anionic surfactant sodium dodecyl benzene sulfonate (SDBS) using native kaolinite, activated kaolinite (AKC) and kaolinite clay mineral composite (KCC) as adsorbent materials at bench scale. Another objective of the study was to determine the impact of mineralogical and physiochemical properties of clay on its capacity to adsorb anionic surfactant. Native, AKC and KCC were successfully prepared by precipitation method. FTIR analysis indicated the presence of functional groups like symmetric/anti-symmetric CH_2, OH with Al^{3+} and free OH. XRD pattern confirmed that different minerals like magnesium silicate hydroxide were discovered after activation of kaolinite. Intra particle diffusion model showed that chemical reaction took place with surfactant molecules on the active site of biosorbent. Kinetic and Isothermal model showed that the chemical reaction followed pseudo second order kinetic and Freundlich model respectively. Adsorption was favored at low pH (2, 3, 4) for native, AKC and KCC, low adsorbent dose (0.5 g) and at low temperature (30°C). The experimental results exhibited the 71% removal of SDBS after only 60 min by KCC. The results suggest that KCC could become excellent candidate for making commercial adsorbent materials.

Keywords: Adsorption; Freundlich model; Kaolinite clay composite; Sodium dodecyl benzene sulfonate; XRD

1. Introduction

Industrialization and widespread use of pesticides in both developing and developed nations have amplified the concerns of pollutants in drinking water [1]. The entry of possibly corrosive and carcinogenic substances into the ecosystem is increasing constantly [2]. Among corrosive chemicals sodium dodecyl benzene sulfonate (anionic surfactant) from detergent industry, household or vehicle washing, food processing, cosmetic, laundry, paper industry and electroplating is a major soil and water pollutant [3]. Metabolites of anionic surfactants with longer half-life (sulfophenyl carboxylate) [4] cause the reduction of oxygen supply by forming the foams in rivers thereby affecting the aquatic lives [5]. These metabolites adsorption onto soil particles also affect its stability, physiochemical characteristics, hydraulic conductivity and biological properties [6]. Environment protection agency (EPA) recommended that safe limits of anionic surfactants for drinking or other purposes is 0.5 mg/L and 1.0 mg/L respectively [7]. According to one study, approximately 40–55 M people do not have access to pure drinking water and around 630 children expire every day due to water borne illness, e.g., diarrhea [8]. As the entry of these anionic surfactants and carcinogenic chemicals into the

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water commodities cannot be prevented, yet development of efficient and cost effective purifying techniques is need of time to ensure water safety [9]. Many reported methods of water purification (removal of surfactants), i.e., chemical precipitation [10], biological techniques [11,12] photocatalysis [13], membranes technology [14] and adsorption [15–18] are inefficient and/or expensive [19]. A new technology using the principle of adsorption for corrosive chemicals like sodium dodecyl benzene sulfonate (SDBS) from wastewater have been explored. It has been reported that adsorption capacity of cationic kaolinite clay and its composites was higher than other synthetic resins [20–23]. There is not a single report presenting the treatment of industrial effluent anionic surfactant SDBS by cost effective and cheaper composites of kaolinite. Kaolinite clay mineral (white clay) is microporous formed by feldspar of aluminum silicate with SiO_4 tetrahedral sheets linked by oxygen atom to octahedral sheets of AlO6 [24]. The exchangeable cations, i.e., Na^+, K^+, Ca^{2+} or Mg^{2+}, etc. Compensate excess of negative charge produced by partial substitution of Si^{4+} by Al^{3+} [9]. Different affinities presented by activated kaolinite and other natural adsorbents depend on the surface area. Anionic exchange and hydrophobic interactions are involved in the adsorption of anionic surfactant onto positively charged kaolinite surface [25,26]. Kaolinite clay mineral exhibits a wide range of adsorption capacity, e.g., when the surfactant concentration is low, monolayer of anion surfactant will be on the surface of kaolinite, while bilayer of surfactant molecule will be attached by hydrophobic interaction to kaolinite at high surfactant concentration [27]. Now kaolinite surface charge has been changed from positive to negative and instantly. After shifting surface charge from +ve to –ve kaolinite exhibits cation exchange capacity [28,29]. Keeping in view the characteristics of kaolinite, this study has been conducted with following objectives: to prepare and compare the efficiency of kaolinite clay mineral, activated kaolinite clay mineral and it's composite to enhance the removal efficiency of sodium dodecyl benzene sulfonate (SDBS, anionic surfactant) from simulated wastewater.

2. Materials and methods

Analytical grade chemicals of Sigma Aldrich Chemical Co. (USA) and Merck (Germany) were used.

2.1. Preparation of kaolinite clay mineral

Kaolinite clay minerals samples (for biosorbent) were collected from different areas of swat district, KPK, Pakistan. To obtain the desired particle size, the solid clay was broken down into smaller pieces before being ground in a food processor (Philips, Japan). The ground material was sieved through an Octagon sieve (OCT-DIGITAL 4527-01) whose mesh size was 300 µm. It was mixed with same particle size rice bran into 3:1 ratio to enhance biosorption capacity [2].

2.2. Activated kaolinite clay mineral (AKC) preparation

Acid-activated kaolinite was prepared by refluxing kaolinite clay for 1 h with 0.5 N H_2SO_4. The resulting activated kaolinite was washed until negative sulphate reaction and dried at 100°C until constant weight.

2.3. Preparation of kaolinite composite (KCC)

KCC was prepared by precipitating iron oxide from a solution of hydrated iron sulphate and iron chloride ($FeSO_4{\cdot}7H_2O$ and $FeCl_3{\cdot}6H_2O$) on kaolinite surface, with constant stirring for 30 min. Iron oxide was precipitated by adding NH_4OH solution. Dark brown suspension was obtained, dried in oven at 60°C. Final product was washed with deionized water. Amount of kaolinite was adjusted to obtain kaolinite/ iron oxide ratio of 4:1 (w/w).

2.4. Preparation of adsorbate solution

A stock solution of 1 g surfactant was prepared in 1000 mL of distilled water. Sub-solutions having the surfactant concentrations from 25 to 200 mg L^{-1} were prepared from the stock solution. Anionic nature of surfactant was confirmed spectrophotometerically at 584 nm.

2.5. Characterization of biosorbents (Kaolinite, AKC and KCC)

Kaolinite, AKC and KCC were chemically analyzed and interpreted by FTIR (Spectrum 2, Perkin Elmer) as KBr discs after sample preparation. The surface structure of kaolinite clay, AKC and KCC were investigated by X-ray diffractometer (XRD, D8 Advance, Bruker).

2.6. Batch study

Parameters like, dose (biosorbent), contact time, initial concentration, pH and temperature should be optimized for maximum adsorption of the surfactant from simulated wastewater. A 50 mL of surfactant solution in 250 mL conical flasks having known concentration, biosorbent dose and pH were shaken in orbital shaking at 121 rpm with control. Following relationship was used to calculate the equilibrium biosorption uptake, q_e (mg g^{-1}):

$$q_e = (C_i - C_e)V/W \tag{1}$$

where C_i is the initial surfactant concentration (mg L^{-1}), C_e is concentration of surfactant at the equilibrium (mg L^{-1}), V is the solution volume (L), and W is the biosorbent mass (g).

2.7. Biosorption equilibrium

Freundlich–Langmuir isotherm models [30,31] were used in equilibrium experiment at different initial concentration of surfactant ranging from 25 to 200 mg/L and with known amount of kaolinite, AKC and KCC in continuous shaking mode in an orbital shaker at 121 rpm keeping temperature constant (30°C).

2.8. Biosorption kinetics

Kinetic models like intraparticle diffusion [32], pseudo-first-order [33] and pseudo second order [34] were determined by adopting continuous shaking mode in an orbital shaker at 121 rpm at constant temperature (30°C) for different time intervals (5–60 min).

3. Result and discussion

Batch study was carried out for testing the capacity of native, activated and composite kaolinite to adsorb an anionic surfactant sodium dodecyl benzene sulfonate (SDBS) from synthetic wastewater of varying concentrations of the contaminant. Equilibrium, thermodynamic and kinetic models were employed to understand the reaction dynamics.

3.1. Effect of pH

During the treatment of industrial wastewater pH is the most important parameter that influences the adsorption capacity of biosorbent as well as the mechanism of adsorption. In our study, we tested the adsorption capacity of kaolinite clays to remove the surfactant at a pH range of 2–10 (Fig. 1). The native, activated and composite of kaolinite showed maximum adsorption capacity of 21.85, 24.98 and 23.00 mg g^{-1} at low pH of 3, 2 and 4, respectively. It has been reported that adsorption increases with decrease in pH [35]. Efficient removal of anionic surfactant at acidic pH might be due to less electrostatic attractions between surfactant anions as a result of insufficiency of cationic sites on kaolinite surface. Positively charged groups of a biosorbent material, increase at low pH thereby providing binding sites for adsorption of anionic surfactant [36,37]. We expect the same may have happened in our study. Bhatti et al. [38] reported that interactions could not be found in solutions but possible at a solid interface.

3.2. Effect of biosorbent dosage

Biosorbent dosage critically influences the biosorption process. To check the effect of biosorbent dose, biosorbents (ranging from 0.05 to 0.3 g/50 mL) were prepared with different forms of kaolinite clay (native, activated kaolinite and composite) at pH 3, 2 and 4 respectively at 30°C temperature. Maximum adsorption of the surfactant was observed against the minimum initial dose of all the sorbent materials, i.e., 0.05 g/50 mL (Fig. 2). With increasing the initial dose of the adsorbent from 0.05 to 0.3 mg/50 mL, the adsorption capacity of composite, AKC and native clays decreased from 35.31 to 14.71 mg/g, 31.85 to11.46 mg/g and 30.69 to 9.04 mg/g respectively. These results are similar to those reported by Bhatti et al. [38]. We argue that the aggregation of particles with increasing initial dose of adsorbents might have decreased the surface area and increased the diffusion path length thereby lowering the adsorption capacity.

3.3. Effect of time

Time is considered an important parameter for designing the low cost and effective methods for the treatment of wastewater. Effect of contact time (5–120 min) of native kaolinite clay, AKC and KCC was determined at optimum pH (2, 3, 4), optimum temperature (30°C) and at (0.05 g) biosorbent dose. The results exhibited that at beginning the adsorption capacity of native, AKC and KCC increased steadily but with the passage of time slowed down until equilibrium was reached (Fig. 3). This might be due to the presence of wide array of vacant surfactant binding active sites in the beginning, which were gradually filled thereby lowering the adsorption speed and eventually leading to establish equilibrium. Banyaladzi and Lisset [39] have also reported that vacant active sites are available initially and with the passage of linear graph is obtained due to filling of active site.

3.4. Effect of concentration

Biosorbent initial concentration is very important to determine the relation between surfactant and number of binding sites on the biosorbent. The effect of initial concentration of biosorbent (25–200 mg/L) on adsorption capacity of native, activated and composite of kaolinite was investigated using optimized initial dose of the sorbent (0.05 g), pH (2, 3, 4) and temperature (30ºC). The results showed that the adsorption capacity of surfactant was dependent on initial concentration of the AKC, KCC and native (Fig. 4). It may be attributed that at initial low surfactant concentration,

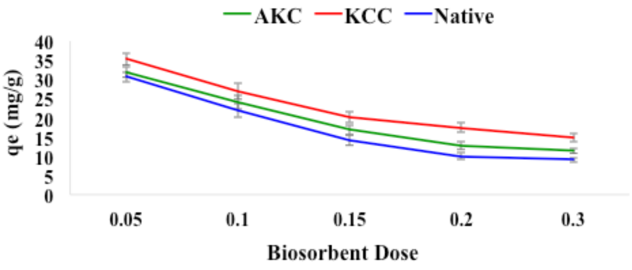

Fig. 2. Effect of biosorbent dose on the biosorption of surfactant by native, AKC and KCC.

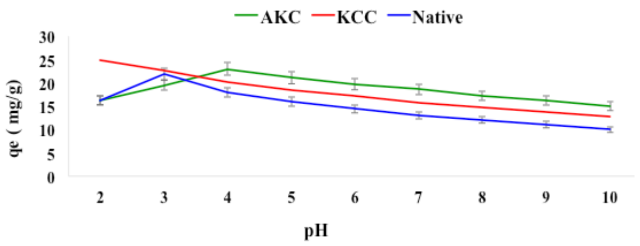

Fig. 1. Effect of pH on the biosorption of surfactant by native, AKC and KCC.

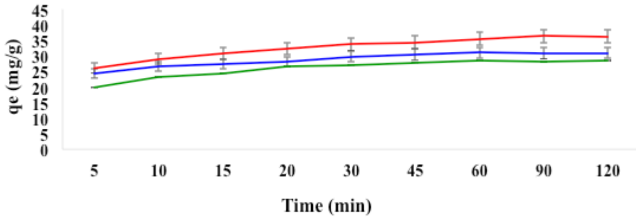

Fig. 3. Effect of time on the biosorption of surfactant by native, AKC and KCC.

vacant active sites were relatively abundant on the biosorbent surface which decreased with increasing number of active sites being filled on increasing the surfactant concentration. These findings were also similar with the findings of Pinhua and Ming [40]. Zhu et al. [41] also suggested that active sites might be closed above the optimal amount of adsorbent.

3.5. Effect of temperature

To determine the nature of adsorption process whether exothermic or endothermic, temperature is considered very important, as most of the chemical reactions in industry require high temperatures [42]. The capacity of AKC, KCC and native to adsorb the surfactant was tested at pH (2, 3, 4), dose (0.05 g), for 30 min at 30–60°C range of temperature. Results revealed that adsorption of surfactant on AKC, KCC and native decreased with increase in temperature (Fig. 5) indicating exothermic nature of the adsorption process. Similar trend has been reported by Kumar et al. [43]. Silvio and Jorg [44] also investigated the adsorptive removal of anionic surfactant sodium dodecyl benzene sulfonate by ZMS and reported that adsorption of anionic surfactant is exothermic in nature.

3.6. Kinetic studies

The kinetic study was performed at different time intervals by keeping anionic surfactant and biosorbent concentration fixed. Kinetics of adsorption by any adsorbent has been commonly tested by pseudo-first-order [33] and pseudo-second-order approach. In the present study, the coefficient of determination (R_2) for the pseudo second-order-model [34] was higher in comparison to pseudo-first-order model, which showed that the kinetic data followed the pseudo-second-order model (Fig. 6)

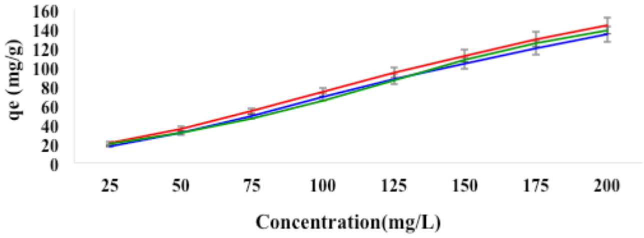

Fig. 4. Effect of concentration on the biosorption of surfactant by native, AKC and KCC.

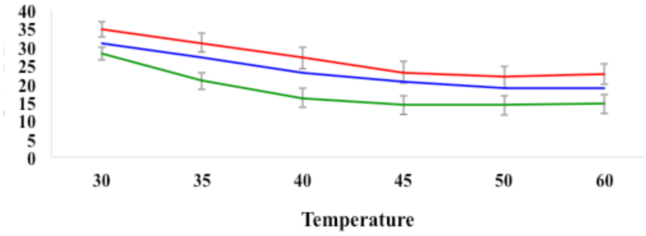

Fig. 5. Effect of temperature on the biosorption of surfactant by native, AKC and KCC.

3.7. Pseudo first order kinetic model

These models represent the relation between amount of surfactant and time. The change in the concentration of surfactant is directly related to value of 1. The differential equation of pseudo first order differential equation is given below.

$$\log\,(q_e - q_t) = \log\,(q_e) - k_1/2.303\ t \qquad (2)$$

3.8. Kinetic model for pseudo second order

Mechanism of results for complete equilibrium range can be explained by pseudo second order. That depends upon adsorption capacity of the biosorbent.

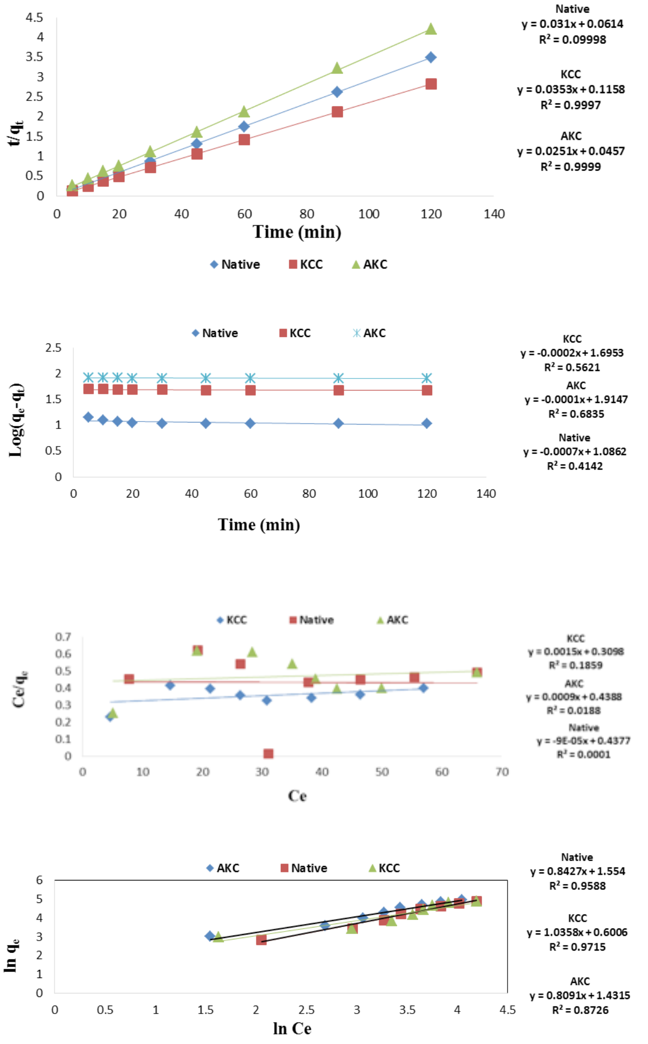

Fig. 6. (a) Pseudo-second order plot for the biosorption of surfactant by native, AKC and KCC, (b) Pseudo-first order plot for the biosorption of surfactant by native, AKC and KCC, (c) Langmuir isotherm model for the biosorption of surfactant by native, AKC and KCC, (d) Freundlich isotherm model for the biosorption of surfactant by native, AKC and KCC.

The equation is given below:

$$t/q_t = 1/\,k_2\,q_e^{\,2} + 1/\,q_e \tag{3}$$

The result showed that the R_2 values for native, AKC and KCC were 0.9998, 0.9999 and 0.9997, respectively. So it is effective and more appropriate than pseudo-first-order kinetic model. These findings are in accordance with the results of Sadaf and Bhatti [45].

3.9. Intraparticle diffusion model

Movement of surfactant molecules towards biosorbent surface involves different steps in the biosorption process. Arrival of molecules at the biosorbent surface is the first step in bulk diffusion. Diffusion of molecules on the other surface boundary of the biosorbent surface following the film diffusion is the second step. However in the 3rd step the molecules will move into the interior pores by intraparticle diffusion from outer boundary. In the last step, surfactant molecules adsorbs on the active sites of biosorbent by chemical reaction. The equation of intraparticle diffusion modal is given below:

$$q_t = K_{\mathrm{pi}}\,t^{1/2} + C_i \tag{4}$$

Here C_i is intercept that explains the thickness of the boundary and it is raised with increase in concentration. The values of C_i suggested that surface diffusion plays role as the rate-limiting the overall sorption process and K_{pi} (mg/g) is the rate constant of intraparticle diffusion. Parameters of intraparticle diffusion model were calculated to determine whether intraparticle diffusion or film diffusion is rate limiting step. The values of the intra-particle diffusion model constants are shown in Table 1. According to this model if the plot of q_t vs $t^{1/2}$ is linear then the biosorption mechanism will be single layer intraparticle diffusion. When the process of biosorption is controlled by more than one mechanism, then a plot of q_t vs $t^{1/2}$ will be multi-linear.

Table 1
Comparative study of kinetic parameters for the biosorption of surfactant

Kinetic models	Native	AKC	KCC
Pseudo-first-order			
K_1 (L min^{-1})	–0.016	–2.3(10^{-4})	–4.6(10^{-3})
q_e (mg g^{-1})	12.19	82.17	49.58
R^2	0.424	0.6835	0.5623
Pseudo-second-order			
K_2 (g mg^{-1} min^{-1})	0.015	0.013	0.01
q_e (mg g^{-1})	32.26	39.84	28.32
R^2	0.9998	0.9999	0.9997
Intra-particle diffusion			
K_{pi} (mg g^{-1} min $^{-1/2}$)	0.7163	0.8213	1.0976
C_i	24.562	21.08	26.304
R^2	0.7368	0.6897	0.8407

The values of co-relation coefficient (R^2) were in the order of KCC > Native > AKC indicating that KCC was the best sorbent having maximum adsorption capacity than native and AKC.

3.10. Biosorption isotherms

The biosorption isotherm reflects interaction between the adsorbent and adsorbate until point of equilibrium is reached [46]. To optimize the efficiency of adsorbent various isotherm models fitted to experimental data.

In this work, the experimental equilibrium data of anionic surfactant were fitted to Langmuir and Freundlich models and values of constants are given in Table 1. The comparison of the R^2 (coefficients of determination) values suggests whether the isotherm equation is valid or not.

3.11. Freundlich isotherm

Freundlich isotherm is based on the fact that the surface of the adsorbent is heterogeneous. With the increase in occupied active sites energy of biosorption logarithmically decreases following the nonlinear equation given as follow:

$$q_e = K_f\,C_e^{\,1/n} \tag{5}$$

Here K_f represents Freundlich constant, determines the surfactant adsorbed on biosorbent per equilibrium concentration whereas n explains the solubility of process. The feasibility of the adsorption of anionic surfactant on AKC, KCC and Native depends upon the value of n and K_f.

Similar findings were obtained by Zhang et al. [36]. Values of q_e revealed that sorption capacity of KCC > Native > AKC which means that KCC has higher biosorption capacity than native and AKC. Preeti and Singh [47] described that presence same minerals in the clay.

3.12. Langmuir isotherm

Langmuir isotherm is based on the fact that the surface of the adsorbent is homogeneous and chemical forces are involved. The simple form of the equation is given below

$$C_e/q_e = C_e/q_m + 1/\mathrm{K_a}q_m \tag{6}$$

Results of Langmuir and Freundlich (Table 1) revealed that Freundlich model is best fitted as compared to Langmiur model.

3.13. FT-IR study

In order to find out the reason behind increased adsorption capacity of activated kaolinite clay mineral (ACC) and kaolinite composite (KCC), FT-IR Study was conducted. FT-IR is an important technique for the spectral analysis of biosorbent surface to identify the distinct functional groups, chiefly liable for the adsorption process. FTIR data confirmed the introduction of functional group into ACC and KCC which increased their adsorption capacity (Fig. 6a,b). The band present between 3701 to

3542 cm^{-1} region is due to OH (stretching) (Fig. 6a). Peak at 3622 cm^{-1} showed the inner layer of OH, which is due to the bonding of OH with Al^{3+}. Davarcioglu and Ciftci [48] and Yleana [49] reported that all peak fall at 3622 and 3626 cm^{-1} respectively. This peak was also confirmed by the findings of Aroke and Nafaty [50]. The peak at 3540 cm^{-1} is due to H–O–H stretching. But Davarcioglu and Ciftci [48] as well as Pinhua and Ming [40] did not observe this peak; rather they observed a peak at 3433 cm^{-1}. This difference in our findings might be due to the amount of water vapors absorbed by the Native (kaolin clay) as a result of its treatment before the spectra were taken. The peak at 3701 cm^{-1} is due to free O–H stretching. The peaks between 2930 and 2820 cm^{-1} showed CH$_2$ symmetric stretching and CH$_2$ anti-symmetric stretching vibrations respectively. When clay is activated the peaks of the OH absorption band shifts from 3660 to 3680 cm^{-1} and 3590 to 3600 cm^{-1} respectively. Furthermore, the peak of the CH$_2$ anti symmetric stretching vibrations shifts from 2930 to 2940 cm^{-1} and for the symmetric vibrations, 2820 to 2830 cm^{-1} respectively. Highly ordered all-trans conformation is characterized by shifting of band to lower wavenumber whereas disorder gauche conformation is characterized by shifting of band towards higher wavenumber. Molecular environment of activated clay can be determined by the frequency shifting of CH$_2$ stretching vibrations. Liquid-like environment of activated kaolinite clay can be inference from higher frequencies while a solid-like environment can be result of lower frequencies.

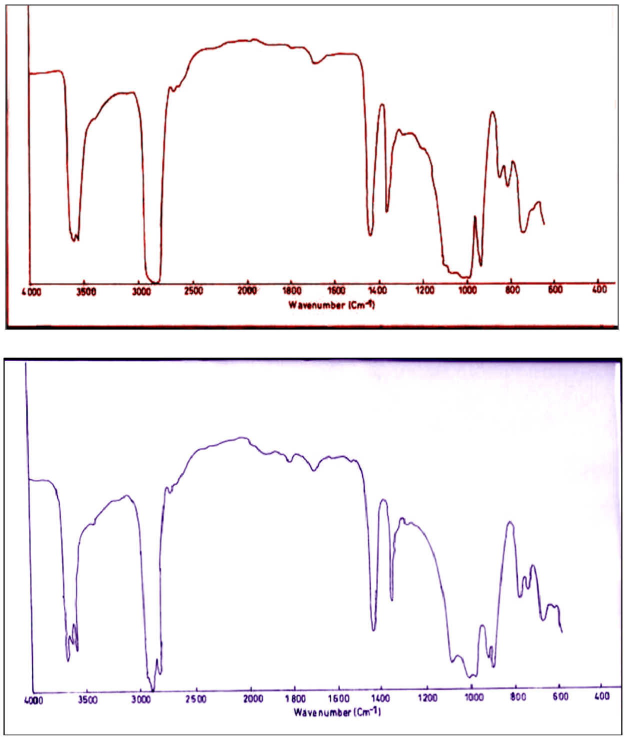

Fig. 7. FT-IR spectrum of (a) kaolin clay (b) AKC.

3.14. XRD analysis

In order to determine the mineralogical composition of the kaolinite (Native), X-ray diffraction analysis of Activated kaolinite clay minerals and kaolinite composite was performed (Fig. 7a–c). Data showed that base minerals: potassium aluminum silicate hydroxide, aluminum silicate hydroxide and aluminum silicate were present in native while sodium aluminum silicate hydrate is present in both native and activated kaolinite clays. After activation, magnesium silicate hydroxide was discovered. Preeti and Singh [47] described that presence same minerals in the clay.

4. Conclusion

Feasibility of clay composites as a promising biosorbent has been highlighted in this study with a noticeable biosorption capacity for the adsorption of sodium dodecyl benzene sulfonate from aqueous solution. Biosorption capacity of kaolinite clay could be enhanced by its activation with H$_2$SO$_4$. It could be concluded form batch experiments conducted with native, AKC and KCC that biosorption process was seemed to be exothermic and rapid in nature. Maximum removal of sodium dodecyl benzene sulfonate with KCC, AKC and native was 71.89, 67.0, and 68.50 mg g^{-1}, respectively. The results revealed that following pseudo- second-order kinetic model and Freundlich adsorption isotherm model, kaolinite clay composites could be applied commercially as a unique bio adsorbent for the removal of surfactant from industrial wastewaters.

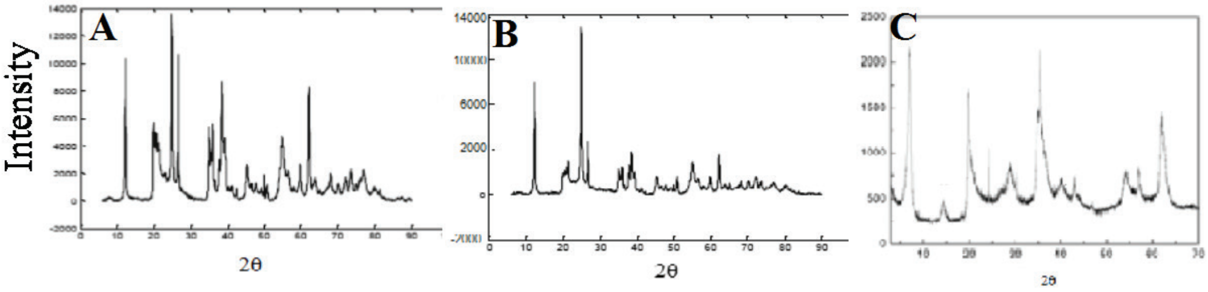

Fig. 8. X ray diffraction pattern for Native (a) AKC (b) KCC (c).

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