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Synthesis and characterization of sugarcane bagasse/zinc aluminium and apple peel/zinc aluminium biocomposites: Application for removal of reactive and acid dyes

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Abstract. In this research work, synthesis of sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite and their application for removal of Reactive Red-241 and Acid Orange-7, respectively, was studied using various parameters. At pH 2 the sorption was the highest for both dyes. The trend showed that the dye sorption declined by decreasing the biocomposite dose and enhanced by increasing the dye concentration and temperature. Equilibrium was achieved at 60 minutes for Reactive Red-241 onto sugarcane bagasse/zinc aluminium biocomposite and 90 minutes for Acid Orange-7 onto apple peel/zinc aluminium biocomposite.The research data was good fitted to pseudo-2nd-order kinetic model and Langmuir isotherm. FT-IR analysis was used to confirm the biosorption of the selected dyes at the surface of biosorbent through various binding sites. Surface morphology modification of both biocomposites before and after biosorption was inspected through SEM. Crystallinity of biocomposite was examined through XRD analysis. It was implied that sugarcane bagasse/ zinc aluminium biocomposite and apple peel/ zinc aluminium biocomposite are good adsorbents for dyes elimination from aqueous solutions.

Keywords: biosorption; sugarcane bagasse/zinc aluminium biocomposite; apple peel/zinc aluminium biocomposite; acid dyes; reactive dyes; modeling

1. Introduction

Water pollution imparts severe effect on health of living beings. The intense use of dyes in the various industries produces hazardous environmental pollution. Different industries like textile, paper, leather and polymer industries use considerable amount of dyes (Safa 2015).

These are highly toxic and carcinogenic in nature. Discharge of these dyes and pigments in water is not only unsafe for aquatic life but also harmful for humans. Various organs such as heart, kidneys, lungs, liver, brain, and central nervous system may be destroyed. Most of them are not easily degraded and stable to light (Safa 2014, 2015). Different procedures are applied for elimination of dyes including physical, chemical and biochemical methods. Surface sorption, chemical precipitation, ion exchange and membrane filtration are incorporated. But these processes have some shortcomings like ineffective dye elimination, expensive and not valid to wide variety of dye waste water. Another disadvantage is formation of other pollutants because of the too much use of chemicals (Bulut *et al*. 2007, Crini 2006).

Biosorption is a proficient and less costly method for the wastewater treatment. Production of sludge is less in biosorption and it is environmental friendly process (Zaheer *et al*. 2014). Various agricultural wastes such as peat and pith (Mittal *et al*. 2005). wheat straw and apple pomace(Ho and Mckay 2003), rice husk (Safa and Bhatti 2011) and waste cellulose (Robinson 2002) have investigated for dyes removal from discarded water. But these biosorbents have mostly low biosorption capacities. So, there is an increasing need to explore new, efficient and easily accessible biosorbents (Safa and Bhatti 2011).

In the present study sugarcane bagasse/zinc aluminium and apple peel/zinc aluminium biocomposites are synthesized and used for removal of reactive and acid dyes. Effects of various features like pH, dye concentration, contact time, pore size of biocomposite, temperature, biosorbent dosage were investigated. Mechanism of biosorption on biocomposite was studied through equilibrium and kinetic data. Characterization of biocomposites is done by FT-IR, SEM and X-Ray Crystallographic analysis.

2. Materials and methods

2.1 Chemicals

All the chemicals used were procured from Sigma–Aldrich Chemical Company, U.S.A.

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Table 1 General characteristics of dyes

Dye	Color	Type	λmax (nm)
Acid Orange 7	Orange	Acidic	410
Reactive Red-241	Red	Reactive	400

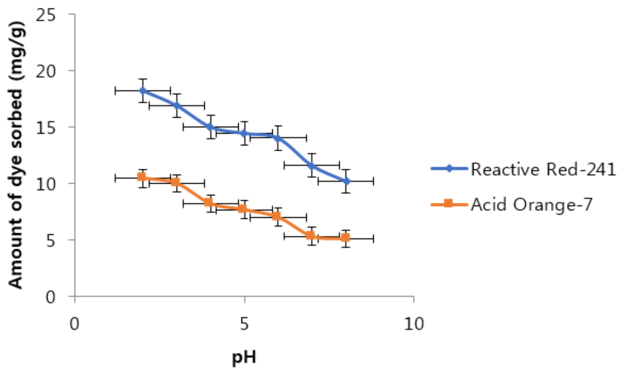


Fig. 1 Effect of pH on the biosorption of Reactive Red-241 and Acid Orange-7 dyes

2.2 Preparation of biocomposite

Sugarcane bagasse and apple peel were collected from market. The biomasses were washed several times with to take out any contaminations. Then, biosorbents were retained in daylight for 5 days and dried at 65°C for 20 hrs. After grinding, separated through standard sieves to get biosorbent of various particle sizes and then preserved them. In order to prepare biocomposite, biomass of small particle size is proceeding further. Sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite were synthesized according to following procedure:

Zinc nitrate (5g) and aluminium nitrate (2g) were dissolved in 100ml of distilled water with vigorous stirring at 85°C for 30 minutes. Then 20 ml of 30% NH_3 solution was added gradually to the solution. Mixture was stirred at 85°C for 2 hours. The synthesized biocomposites were washed away with distilled water and dried under vacuum.

2.3 Dyes solution preparation

In the research, Reactive Red-241 and Acid Orange-7 dyes (took from market) were used. Stock solution of both dyes, were prepared. These stock solutions were used for the preparation of working solutions of various concentrations by appropriate dilution.

Features of two dyes are shown in Table 1.

2.4 Experimental biosorption studies

Batch biosorption tests were done to determine the influence of factors like dye concentration, pH, contact time, temperature, particle size and biocomposite dose on the dyes removal. The amount of absorbed dye was determined through the equation:

$$q = (C_o - C_e) \times V/W \quad (1)$$

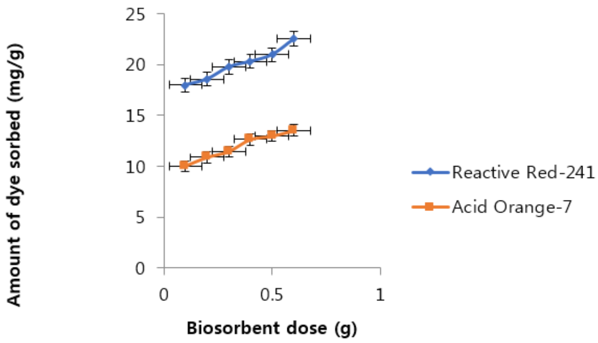


Fig. 2 Effect of biosorbent dose on the biosorption of Reactive Red-241 and Acid Orange-7 dyes

q in units of mg/g represents sorption capacity of the biocomposite for the dye, Co is the initial and Ce is the equilibrium dye concentrations. V, is the volume of dye solution in ml, M is the quantity of biocomposite (g). The sorption percentage was determined as follows:

$$\% \text{ sorption} = C_o - C_e/C_o \times 100 \quad (2)$$

2.5 Studies of Sorption isotherm

Biosorption is determined through sorption isotherms. Two models were applied on the data such as the Langmuir (1916) and Freundlich (1906) isotherms.

2.6 Studies of Sorption kinetics

The pseudo 1st order (Lagergren 1898) and pseudo 2nd order (Ho *et al*. 2000) models were used to evaluate the performance of dyes onto the sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite.

3. Results and discussion

3.1 Impact of pH

The biosorption of a dye, significantly rest on the solution pH. Solution pH affects the biocomposites, influences the biosorption process and the ionization of the dye molecules. Earlier experiments on dye elimination have shown that ionization and surface charge of dyes are mainly influenced by PH. If the pH of solution varies, the biosorption ability may affect (Önal 2006). To determine the ideal pH of the Reactive Red-241 and Acid Orange-7 dyes, experiments were conducted from 2 to 8. The observations are presented in Fig. 1. The graph shows that dye biosorption was decreased by increasing the solutions pH. At a pH 2 for Reactive Red-241 and Acid Orange-7 dyes, the biosorption is high.

More dye biosorption on each biocomposite at minimum pH may result from the counterbalancing of surface negative charge of the biocomposite, so increasing the protonation. This accelerates diffusion and biosorption capacity increased due to active surface of the biocomposites (Gupta 2005) Sathishkumar *et al*. (2012) also

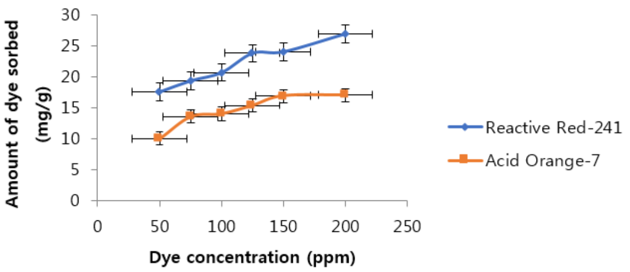


Fig. 3 Effect of dye concentration on the biosorption of Reactive Red-241 and Acid Orange-7 dyes

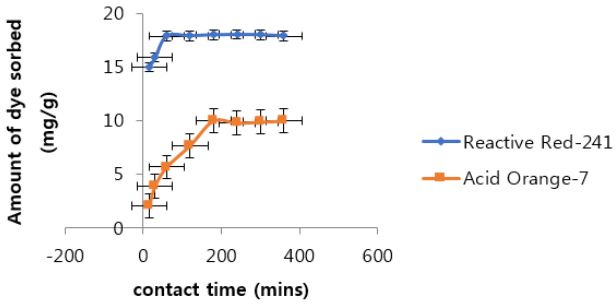


Fig. 4 Effect of contact time on the biosorption of Reactive Red-241 and Acid Orange-7 dyes

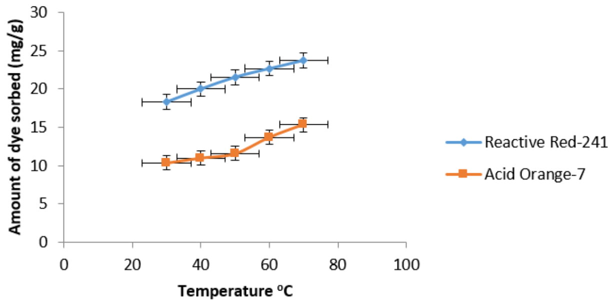


Fig. 5 Effect of temperature on the biosorption of Reactive Red-241 and Acid Orange-7 dyes

investigated a fall in the biosorption capacity of Jatropha curcas by Blue R dye, by increasing solution pH. Normally, cationic dye removal percentage decreases at small pH as compared with anionic dyes. Aksu and Isoglu (2006) observed the pH influence on the biosorption of Gemazol dye by using the pulp of sugar beet and they detected the highest sorption at low pH.

3.2 Impact of the biocomposite dose

The influence of biocomposite dosage on the removal of Reactive Red-241 and Acid Orange-7 dyes on the sugarcane bagasse/zinc aluminium and apple peel/zinc aluminium biocomposites was determined by using different doses of biocomposite and the results are described in Fig. 2. It is evident from the graph that the dye sorption increases with a corresponding rise in the biocomposite dose. The dye sorption capacity was increased from 18.00 to 22.44 mg/g for Reactive Red-241 dye onto sugarcane bagasse/zinc aluminium biocomposite and from 10.01 to 13.54mg/g for Acid Orange-7 dye onto apple peel/zinc aluminium biocomposites. Dye elimination increases with increasing biocomposite dose, because adsorption sites also increase (Ofomaja 2008) and resultantly, the percentage of dye elimination from the solution also enhances.

Sonawane and Shrivastava (2009) investigated the biosorbent dose influence on the Malachite green elimination by maize cob. They showed that the removal percentage was increased with an increase in the biomass dose. In one more research, Bhattacharyya and Sharma (2004) stated the influence of biomass dose on the elimination of Congo red dye. The dye removal was the highest at low dose. Akhtar *et al*. (2006) studied the influence of dose of biosorbent on 2, 4 dichlorophenol uptake. The percentage biosorption enhanced with more biomass dosage.

3.3 Impact of concentration of dye

Dye concentration influences the dye removal. There is a close relationship between dye concentration and sorption sites on a biocomposite surface. The influence of concentration of Reactive Red-241 and Acid Orange-7 dyes on the biosorption ability of biocomposites was investigated from 50 to 200 ppm concentration range. The data is presented in Fig. 3. The graph shows that there is a direct proportion between the dye biosorption and dye concentration. The quantity of Reactive Red-241 biosorbed increases from 17.54 to 26.89 mg/g on the sugarcane bagasse/zinc aluminium biocomposite and from 10.01 to 16.99 mg/g for Acid Orange-7 dye onto the apple peel/zinc aluminium biocomposite. Mostly, the dye removal ability increases with high dye concentration (Eren and Acar 2006).

In 2009, Ergene *et al*. investigated the elimination of RBBR by sorption on Scenedesmus quadricauda. Their results show a high increase in dye elimination with more dye concentration. Mahmoud *et al*. (2012) also observed the similar trend.

3.4 Impact of contact time

It was observed that with the increase in contact time, the biosorption capacity increases rapidly. But further increase shows no remarkable change. It is shown in Fig. 4. The equilibrium biosorption time for Reactive Red-241 dye is 60 min and for Acid Orange-7 dye is 180 minutesand after that no major increase in biosorption occurs. This is attributed to strong interaction between the biocomposite and the dye. Tan *et al*. (2010) observed that the equilibrium time for direct red-23 dye was 30 minutes, the elimination rate is high in the beginning. The removal of congo red dye also increases with time (Namasivayam and Kavitha 2002).

3.5 Impact of temperature

The temperature of discharge industrial water is mostly high. Fig. 5. shows the temperature effect of Reactive Red-241 and Acid Orange-7 dyes onto biocomposites. The results describe that as temperature rises from 30 to 70°C

Table 2 Comparison of the kinetic parameters for the biosorption of Reactive Red-241 and Acid Orange-7 dyes onto sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite

Kinetic models	Reactive Red-241	Acid Orange-7
Pseudo-first-order		
k_1 (1/min)	0.07301	0.0044
q_e (experimental)	18.057	10.552
q_e (calculated)	1.056	0.447
R^2	0.475	0.382
Pseudo-second-order		
k_2 (g/mg min) 10^{-3}	0.1688	0.0851
q_e (experimental)	18.057	10.552
q_e (calculated)	1.254	2.101
R^2	0.995	0.985

the removal capacity of biocomposite also increases. An increase in temperature also causes the motion of dye molecules to increase (Senthilkumaar *et al*. 2006) Moreover, an increase in temperature leads to a corresponding increase in quantity of dye adsorbed due to enhanced mobility of the large dye molecules (Aksu and Tezer 2005).

Mahmoud *et al*. (2012) reported that the temperature increases the removal of basic dye.

3.6 Biosorption kinetic models

In this research, two kinetic models (pseudo-1st-order and pseudo-2nd order) are used to study the biosorption kinetics.

3.6.1 Pseudo-1st-order kinetics

Pseudo-1st-order kinetics describes a change in the dye concentration with time. The differential equation is shown as

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (3)$$

where qe and qt in units of mg/g are biosorption capacities at equilibrium and time t respectively, k1 (in units of min-1) represents the rate constant.

On integrating the above equation for the boundary conditions of t = 0, to t = t and qt = 0 to qt = qt, the above equation takes the form

$$\log (qe/qe - qt) = (k1/2.303)t \quad (4)$$

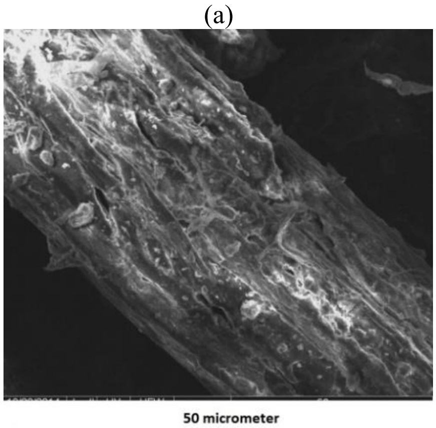
After rearrangement

$$\log (qe - qt) = \log qe - (k1/2.303)t \quad (5)$$

The data for qe calculated, qe experimental, k1 and R2 of both the dyes are presented in Table 2.

The results describe that no relation was found between the experimental and calculated values of qe and the R2 value was also non- acceptable for both the dyes. So, pseudo-1st-order model was not fitted well.

(a)



(b)

Fig. 6 SEM photographs of dye loaded sugarcane bagasse/zinc aluminium biocomposite

3.6.2 Pseudo-2nd-order kinetics

Pseudo-2nd-order kinetics described the biosorption process over a wide range. The equation for pseudo-2nd-order kinetics is given as:

$$dqt/dt = k2(qe - qt) \quad (6)$$

where k2 is rate constant (g/mg min) for the 2nd-order biosorption process, qt and qe represent the capacities of biosorption at time t and equilibrium respectively.

On integration with in the boundary limits of qt = 0– qt = qt and, t = 0– t = t the linearalized form of above equation for 2nd order reactions may be written as:

$$t/qt = 1/K2q2e + 1/qe\ (t) \quad (7)$$

The results are provided in Table 3. The data evidenced that qe and R2 values for both the dyes are satisfactory and the model thus well fitted the kinetic data.

Bulut and Aydin (2006) used the wheat shells to study the sorption of MB and found that second order model is good fitted. Senthilkumaar *et al*. (2006) studied the adsorption of MB by using the guava leaf powder. The R2 - value for the second-order model was 0.999.

3.7 Adsorption isotherms

Adsorption isotherms elaborate the interaction of adsorbent with adsorbate. This work used Langmuir and Freundlich isotherms to study the equilibrium of biosorption process.

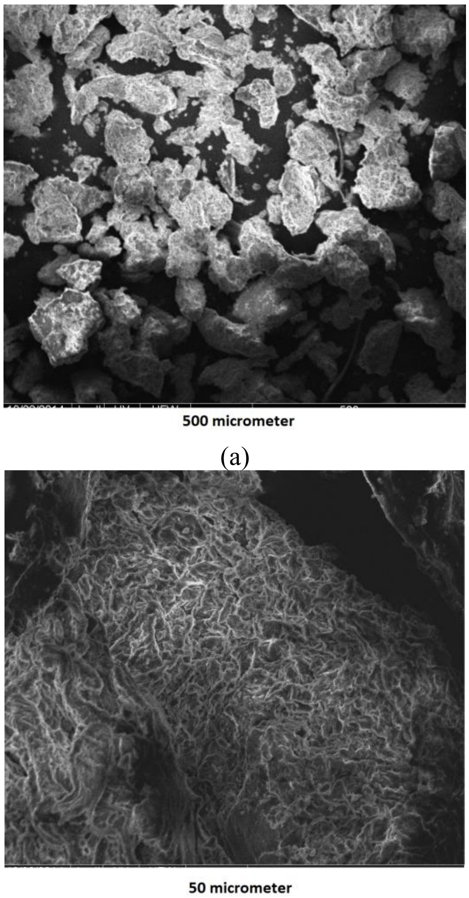


Fig. 7 SEM photographs of dye loaded apple peel/zinc aluminium biocomposite

Table 3 Comparison of the isotherm parameters for the biosorption of Reactive Red-241 and Acid Orange-7 dyes onto sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite

Isotherm models	Reactive Red-241	Acid Orange-7
Langmuir		
R_L	0.213	0.300
R^2	0.987	0.993
q_m	30.113	8.410
Freundlich		
K_F	4.210	1.745
R^2	0.519	0.766
N	0.411	0.257

3.7.1 Langmuir isotherm

Langmuir isotherm is used to study the process of contaminants biosorption from aqueous solutions. The adsorption on the surface may occur either in a monolayer or a multilayer. Langmuir isotherm is an ideal model based on monolayer adsorption (Dabrowski 2001). It is represented by the following equation:

$$Ce/qe = 1/Kqm + Ce/qm \quad (8)$$

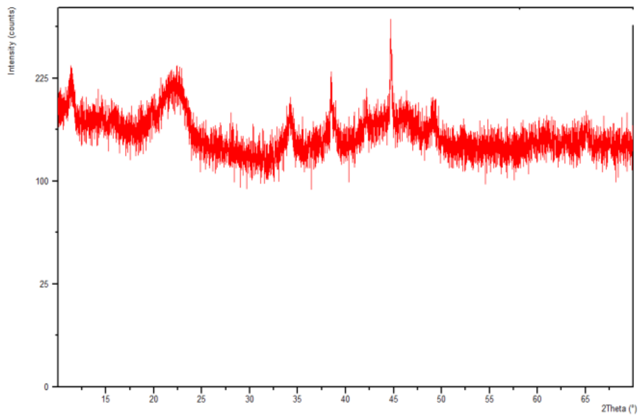


Fig. 8 XRD pattern of dye loaded sugarcane bagasse/zinc aluminium biocomposite

Ce here represents the dye solution concentration (mg/L) at equilibrium, qm in units of mg/g is the adsorption capacity and K is linked with the adsorption energy (L/mg). Values of qm and K are shown in Table 3.

3.7.2 Freundlich isotherm

This basic principal of this model is that the biosorption occurs when the dye molecules interact with the heterogeneous surfaces. Mathematically, the Freundlich isotherm is represented as:

$$qe = Kf\,(Ce)1/n \quad (9)$$

where qe is the quantity in mg adsorbed per gram of the adsorbent, Ce represents the adsorbate concentration at equilibrium in mg/L, and n, Kf, are the Freundlich constants for adsorption intensity, and capacity respectively. The log form of Eq. (9) gives the following linearized expression:

$$\ln qe = \ln KF + 1/n \ln Ce \quad (10)$$

The data for Freundlich constant n is provided in Table 3 which shows that the model is best fitted. Ozacar and Sengil (2003) also studied that the capacity of calicinated alunite for the removal of reactive dyes followed the 2nd-order kinetics.

3.8 FT-IR Studies

The FT-IR spectra of sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite were recorded before and after biosorption of Reactive Red-241 and Acid Orange-7 dyes in the frequency range of 400–4000 cm−1. The spectra showed that the functional groups exchanged the sites of biosorption (Kim *et al.* 2015). The biocomposites spectra exhibited bands at 2924.09 cm−1 for C–H stretch that evidenced the presence of =CH and –CH groups which show that sugarcane bagasse and apple peel contain lignin and these outcomes are very similar to described results (Pavan *et al.* 2008). The peaks for stretching C=O vibrations appeared at 1728.22 cm−1. The vibrations of –OH functional groups were recorded at 3062.96 cm−1. The appearance of these peaks is due to –OH and C=O vibrations that evidences the presence of -COOH group on the biocomposites surface (Yun *et al.*

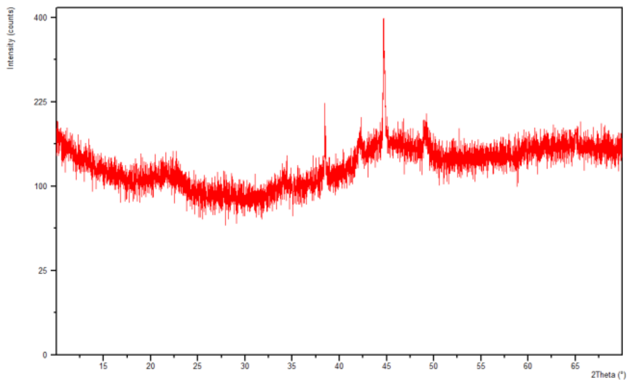


Fig. 9 XRD pattern of dye loaded apple peel/zinc aluminium biocomposite

2001). The existence of –CONH2 (amide group) on the surface of biocomposite was confirmed by the presence of peak at 1633.33 cm−1 and the bifurcation of peak at 3651.25 and 3637.75 cm−1 shows the existence of (Tian *et al*. 2010). The interaction between the biocomposite and dyes molecules lead to certain modifications in the FTIR spectra in the form of either broadening or disappearance of some peaks. In case of dyes loaded biocomposite, the –OH stretching vibrations either disappear or are shifted to lower frequency which indicates the role of -OH and -COOH groups in the sorption process. The involvement of amide group in dye biosorption was confirmed by a decrease in the intensity of –NH peak at 1676.14 cm−1 disappearance of bifurcation at higher frequency.

3.9 SEM Analysis

SEM is the technique used to illustrate the morphology and surface characteristics of the materials such as biosorbents. It defines the porosity of the biomass as well as the shape of particles (Tian *et al*. 2010).A higher porosity confers a higher biosorption potential to the surface of biomass (Arivalagan *et al*. 2014) Figures 6a-6b and 7a-7b depict the morphology of dye loaded sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposit respectively. A white monolayered molecular cloud was formed after the biosorption of reactive Red-241 and Acid Orange-7 dyes. The monolayered coverage was confirmed by studying Langmuir adsorption isotherm in batch manner. Kim *et al.* (2015) determined morphology of the surface of dye treated and untreated biomass consisting of bacillus catenulatus. The presence of dye molecules was confirmed on the surface of biomass after biosorption.

3.10 XRD Analysis

Crystalline nature of compounds was determined by using a proficient technique known as XRD. The crystalline substances exhibited well-defined and sharp peaks while amorphous materials produced diffused peaks. XRD pattern of dye loaded biocomposites (sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite) shows diffused peaks (Fig 8, 9). The XRD pattern of biosorbed Reactive Red-241 and Acid Orange-7 dye showed the change of sharp peaks to the diffused peaks that evidenced that after biosorption the crystalline biomass was transformed into amorphous one. Zolgharnein *et al.* (2013) reported that Carpinus betulus showed well-defined peaks at 2 θ = 38.32, 31.8 and 50.36 were changed to diffused ones after biosorption of phenol and dyes.

4. Conclusion

Removal of Reactive Red-241 and Acid Orange-7 dyes from aqueous solution by biosorption onto sugarcane bagasse/zinc aluminium biocomposite and apple peel/zinc aluminium biocomposite was determined experimentally.

The percentage removal of dyes was affected by various process parameters. Increase in the temperature enhances the biosorption rates. Equilibrium time was observed to be 60 minutes for Reactive Red-241 dye and 90 mins for Acid Orange-7 dye. Biosorption was highest at pH 2. The increase in the adsorption potential with the increase in the biocomposite dosage was attributed to the increase in the number of sorption sites at the biosorbent surface. High temperature increases the biosorption capacity because the motion of dye particles increases. Pseudo-2nd order kinetic model and the Langmuir isotherm are well fitted to data. Characterization of biocomposites was done through FT-IR, SEM and XRD analysis.

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CC

List of Abbreviations

RBBR	Remazol Brilliant Blue R
MB	Methylene Blue
Ppm	Parts per million
Min	minute
FT-IR	Fourier transform Infrared spectroscopy
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction