



# Biosorption of basic yellow dye using leaf sheath powder of *Areca catechu* (Betel nut tree)

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## Abstract

Increased anthropological activities have led to the random spread of pollutants in the environment. The pollutants that enter the environment have the potential to bind up with the elements in the surroundings and form both reversible & irreversible chemical compounds that pose hazards to humans and other living organisms. The availability of treatment and purification techniques of such pollutants is wide spread and of high cost. Our aim is to provide an eco friendly and cost effective material for dye removal. The biosorption of basic yellow dye by the raw and unmodified leaf sheath powder of *Areca catechu* (betel nut tree) was analyzed in this study.

**Keywords:** Biosorption, basic yellow dye, eco friendly method, adsorption isotherms, equilibrium kinetics.

## Introduction

The effluents and untreated water from the industries pose a critical problem to the environment. Thus it is the crucial time to focus more on the activities of mitigating and eradicating ecosystem damage and environmental pollution. The dyes are designed to be chemically and photolytically stable and are hazardous from the ecological point of view. The effluents produced are relatively heavily coloured, contain high concentrations of salt and exhibit high BOD / COD values. Dyes, even at very low concentrations, reduce wastewater transparency and oxygen solubility and are often toxic, carcinogenic or mutagenic for various organisms and recalcitrant.

The treatment of effluents containing dyes has been realized by several methods such as ultrafiltration through membranes, electrochemical, coagulation / flocculation, biological degradation, and photocatalytic degradation. Most of these methods require high capital costs which are not suitable for small-scale industries but the adsorption process is found to be the most efficient, inexpensive, and economically viable. Over the last decades, various materials have been developed as adsorbents for dyes removal from natural waters or industrial wastewaters, but high cost and complex preparation conditions usually limit their practical promotion and application. Recently, low-cost adsorbents developed from natural materials or certain waste materials (or by products) from agricultural or industrial activities such as eggshell waste, hen feather, de-oiled soya, apple pomace, wheat straw, orange peel, banana peel, maize cob, maize stalk, rice husk, wood chip, saw dust, coir pith and aquatic plants have gained greater attention due to many obvious advantages such as easy availability, comparable efficiency, resource generation, and pollution abatement<sup>1</sup>.

In our work, the biosorption of basic yellow dye from its aqueous solution by using *Areca catechu* was investigated. The biosorption capacity of adsorbent was investigated using batch experiments and the influence of contact time, initial dye concentration, adsorbent dosage, solution pH and operational temperature were analyzed and the experimental data obtained were evaluated and interpreted using adsorbent equilibrium isotherms and kinetic models.

## Materials and Methods

**Preparation of biosorbent:** The biosorbent was prepared from the leaf sheath of *Areca catechu* (betel nut tree), the leaf sheath was cut into pieces and dried under sunlight for 4 hours for 3 days. The dried pieces were grounded into coarse powder which is then sieved and used for the experimentation.

**Preparation of experimental solution and chemical reagents:** The stock solution was prepared by diluting 1mg of basic yellow dye in 100ml of distilled water which was diluted in necessary proportions for the experimental solution. 0.1N of Hydrochloric acid and 0.1N of Sodium hydroxide were prepared for optimization of the reaction parameters.

**Characterization:** Morphological characteristics of the biosorbent (leaf sheath powder of betel nut tree) were analysed by Scanning Electron Microscopy (SEM)<sup>2</sup>. In the adsorption studies, the surface characteristics of the adsorbent have a great influence over the process and efficiency of biosorption.

**Batch Biosorption Procedure:** Stock solutions were prepared by dissolving desired amount of dye in distilled water and necessary concentrations were obtained by dilution. The biosorption experiments were carried out in a series of conical

flasks containing 50ml of the synthetic dye solution at desired concentration and desired weight of the biosorbent. The dye solution with the biosorbent was stirred for 3 hours at 300rpm using an electronic shaker. The influence of biosorbent dosage was analysed by varying the range from 50mg/l to 500mg/l. The pH was varied from 2-14 (the basic yellow dye possesses a higher alkaline nature and could be adsorbed easily at basic conditions) at initial dye concentration of 300mg/l. The pH of the solutions was adjusted with either 0.1M of NaOH or 0.1M of HCl. The contact time was varied between 15 and 240 minutes at room temperature was tested from 20°C to 45°C using a thermostatically controlled incubator. The concentration of the dye was varied from 100mg/l to 600mg/l. After biosorption, the solid phase was separated from the liquid by centrifugation at 300 rpm for 5 minutes.



Figure-1: Dried Coconut Leaf Sheath.



Figure-2: Leaf sheath powder.

The biosorption efficiency was calculated using the equation given below,

$$\text{Adsorption Efficiency (\%)} = \frac{C_e - C_0}{C_0} \times 100 (\%)$$

Where:  $C_0$  - Concentration of dye at the initial time,  $C_e$  - Concentration of dye at equilibrium time.

## Results and discussions

**Characterization:** The infrared spectra of the adsorbent before and after biosorption of the basic yellow dye are tabulated below.

**Morphology of the biosorbent:** The surface texture and morphology of the adsorbent was analysed using SEM (Scanning Electron Microscope) in order to study and compare the morphology of biosorbent before and after biosorption. The results showed that the process of adsorption took place in the surface of leaf sheath powder as the surface of the adsorbent after adsorption seems to be smoother than the surface before adsorption<sup>3</sup>.

**Biosorption Performance: Effect of Adsorbent Dosage:** In the process of biosorption, the optimization of the adsorbent dosage is the most essential step which has the ability to determine the efficiency of adsorbent in the removal of dye. Figure-5 represents the removal efficiency of the adsorbent at different dosage of the adsorbent.

**Biosorption Isotherms:** The biosorption isotherms describe how the sorbate molecules are distributed between the liquid phase and solid phase when the system reaches the equilibrium. The analysis of isotherm data by fitting them to different models is important to find a sustainable model that can be used. The biosorption isotherms are illustrated in the Figures-6 and 7. It is obvious that the amount of dye biosorbed increases as its equilibrium concentration increased. This trend may be due to the high driving force for mass transfer at a high initial heavy metal concentration. In addition, if the dye concentration in solution is higher, the active sites of biosorbents are surrounded by more dye molecules and the biosorption phenomenon occurs more efficiently. Thus, biosorption amount increases with the increase of initial ion concentration.

Several biosorption isotherms can be used to correlate the biosorption equilibrium in dye biosorption on several biosorbents. Some well known isotherms used for determining the interaction of the biosorbent with the biosorbate are Langmuir and Freundlich models<sup>2</sup>.

**Langmuir Model:** Langmuir model is based on the assumption that maximum adsorption corresponds to monolayer formation of adsorbate layer on the adsorbent surface.

**Freundlich Model:** Freundlich isotherm is an empirical equation assuming that the biosorption process takes place on heterogeneous surfaces, and biosorption capacity is related to the concentration of biosorbed metal ions at equilibrium.

The  $R^2$  values obtained using the Freundlich isotherm model were lower than those obtained using the Langmuir isotherm model, with  $R^2$  value of 0.996. The perfect fit of the experimental data with the Langmuir isotherm model indicated that the adsorption behaviour was predominantly monolayer adsorption.

**Effect of solution pH:** The pH of the dye solution has a major impact on the rate of biosorption. In this experiment, the pH range was chosen from 2 to 14. The biosorption of the basic

yellow dye was observed with an initial dye concentration of 300mg/l and adsorbent dosage of 350 mg/l for a period of 180 minutes. Maximum biosorption efficiency was attained at the pH of 10 since the dye exhibit basic characteristics. The relationship of biosorption efficiency with varying pH is shown in the Figure-8.

**Table-1:** Shows broad absorption band for the leaf sheath powder of areca nut before and after the biosorption.

Absorption ( $\text{cm}^{-1}$ )		Bond/Functional Groups		Transmittance (%)	
Before Adsorption	After Adsorption	Before Adsorption	After Adsorption	Before Adsorption	After Adsorption
3718.76	3780.48	O-H stretching	O-H stretching	101.73	102.51
3394.72	3703.33	O-H stretching	O-H stretching	99.80	101.98
2978.09	3626.17	CH stretching	O-H stretching	97.07	101.54
2916.37	3348.42	NH stretching	N-H stretching	97.89	101.23
2360.87	3217.27	$\text{C}\equiv\text{N}$ stretching	O-H stretching	92.72	101.37
1728.22	2978.09	C-H binding	C-H (alkanes)	100.96	96.81
1604.77	2916.37	$\text{C}\equiv\text{C}$ stretching	C-H (alkanes)	100.36	98
1465.90	2846.93	C-H binding	C-H (alkanes)	99.50	100.05
1234.44	2661.77	C-N stretching	Phospine	97.80	101.92
1026.13	2360.87	C-N stretching	Phospine	91.74	93.27
894.97	2337.72	$\text{C}=\text{C}$ bending	Phospine	98.13	94.83
763.81	1882.52	C-H bending	C-H bending	96.42	102.79
671.23	1720.50	C-Cl stretching	$\text{C}=\text{N}$ stretching	93.77	102.56
586.36	1658.78	C-Cl stretching	N-H stretching	96.30	102.08
555.50	1597.06	C-Cl stretching	N-O stretching	96.73	101.24
501.49	1535.34	C-Cl stretching	C-H bending	96.71	102.07
	1465.90		C-H bending		100.22
	1381.03		C-O stretching		98.86
	1234.44		hydrate		99.68
	1157.29		S-O stretching		97.35
	1064.71		$\text{C}=\text{C}$ stretching		96.57
	956.69		C-Cl stretching		97.34
	817.82		C-Cl stretching		99.94
	763.81		C-Cl stretching		97.56
	671.23		C-Cl stretching		96.95
	609.51		C-Cl stretching		98.98
	570.93		C-Cl stretching		98.01

Fourier Transform Infrared Spectrum showed several peaks at  $2978.09\text{cm}^{-1}$ ,  $2916.37\text{cm}^{-1}$ ,  $2846.93\text{cm}^{-1}$ ,  $1535.34\text{cm}^{-1}$ ,  $1465.90\text{cm}^{-1}$  for C-H (alkanes),  $817.82\text{cm}^{-1}$ ,  $763.81\text{cm}^{-1}$ ,  $671.23\text{cm}^{-1}$ ,  $609.51\text{cm}^{-1}$ ,  $570.93\text{cm}^{-1}$  for C-Cl (halo compound). This result proves the biosorption of the basic yellow dye onto the surface of the leaf sheath powder.

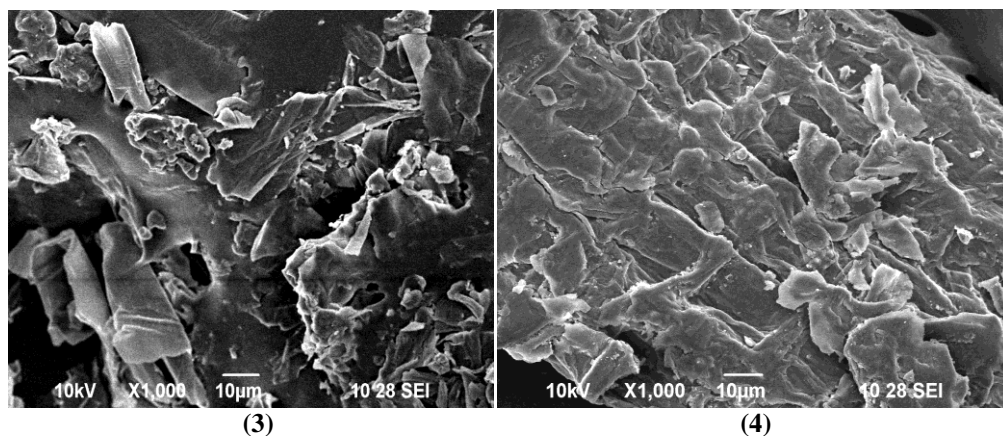


Figure-3 &4: Scanning Electron Microscopy images of Leaf sheath powder of *Areca catechu*.

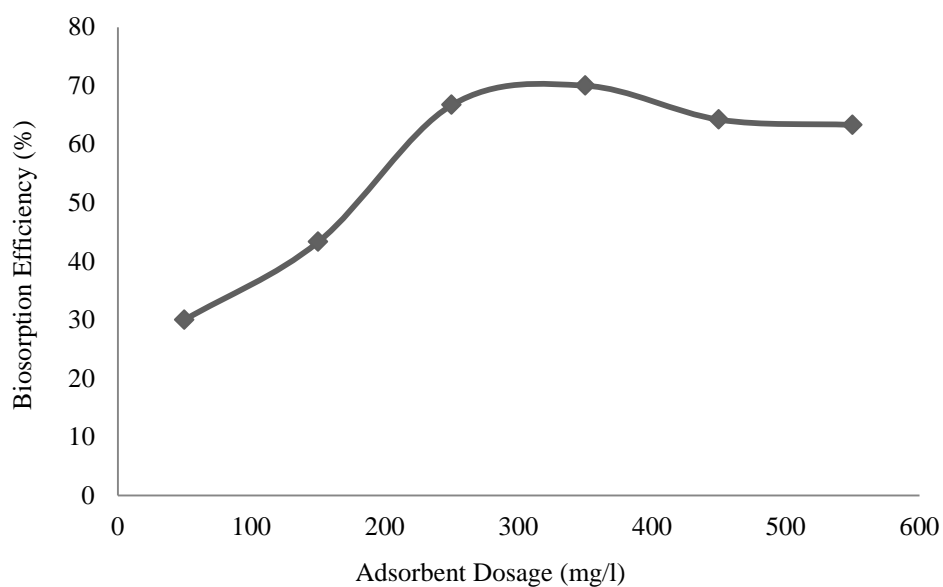
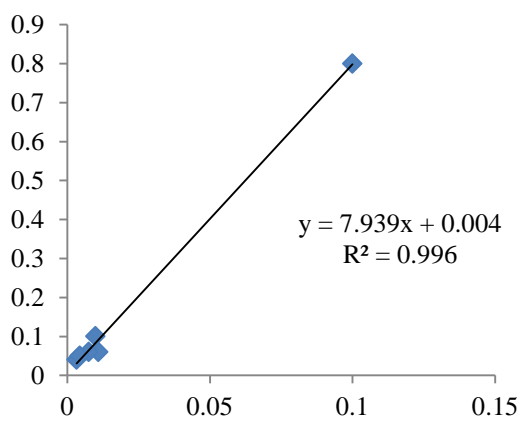
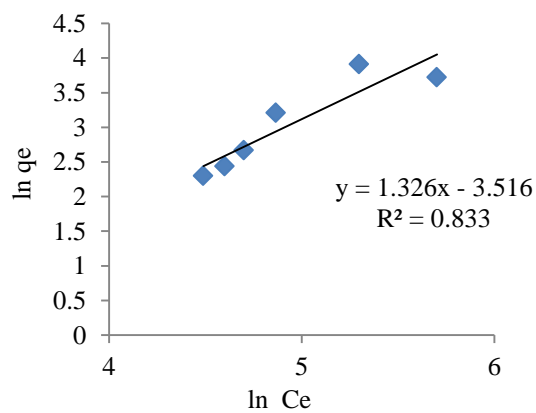


Figure-5: Effect of adsorbent dosage on biosorption efficiency.



(6)



(7)

Figure-6 & 7: Langmuir and Freundlich Isotherm Models.

**Effect of Dye concentration:** The adsorption efficiency of the adsorbent was determined by the amount of dye it had adsorbed from the dye solution. Hence it becomes essential to optimize the dye concentration to evaluate the efficiency of the adsorbent. In this experiment varying concentration of dye was taken from 100mg of basic yellow dye per litre of water to 600mg of basic yellow dye per litre of water. In addition the adsorbent dosage of 350mg/l and pH of 10 were taken as constant parameters for this batch process with a reaction time of 180 minutes. The readings were observed and plotted as graph. From the graph we could infer that the biosorption efficiency was found to be high at 300mg/l of dye concentration.

**Effect of Temperature:** Temperature has a major influence on the functioning of the biological operating systems. Hence it becomes essential that the temperature of a process is optimized for obtaining maximum efficiency. In this experiment, three different temperatures were taken for the optimization process.

The biosorbent dosage, pH of the solution and the initial dye concentration were kept as constant with a reaction time of 180 minutes. It is found that the biosorption efficiency increased with increase in temperature as shown in the Figure-10.

**Effect of rotations per minute:** The effect of rpm on the rate of biosorption was calculated with constant values of adsorbent dosage (35mg/l), pH (10), initial dye concentration (300mg/l) and temperature (35°C). With these parameters, the biosorption efficiency was found to be the maximum at rpm of 200 rotations per minute for a contact time of 180 minutes.

**Effect of contact time:** The duration for which the biosorbent remains in contact with the dye solution determines the rate of efficient removal of the dye and it can be extended until the biosorption attains equilibrium. In this experiment, the equilibrium was attained within 90-120 minutes of the batch reaction which can be understood from the Figure-12.

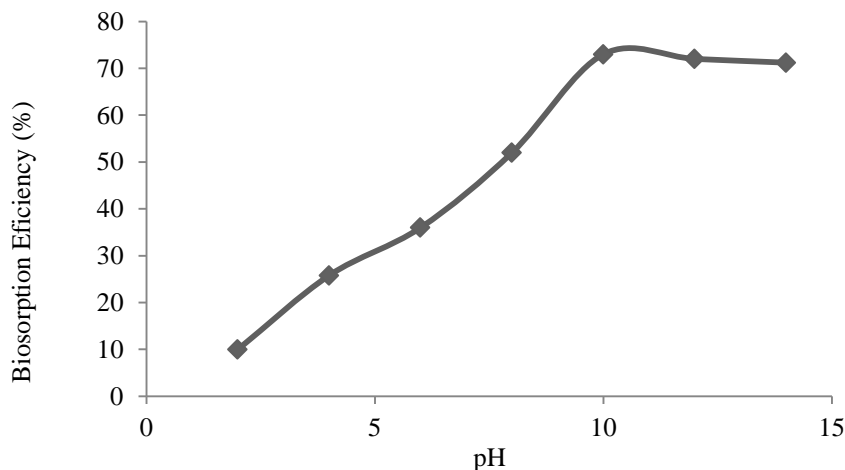


Figure-8: Effect of pH on Biosorption Efficiency.

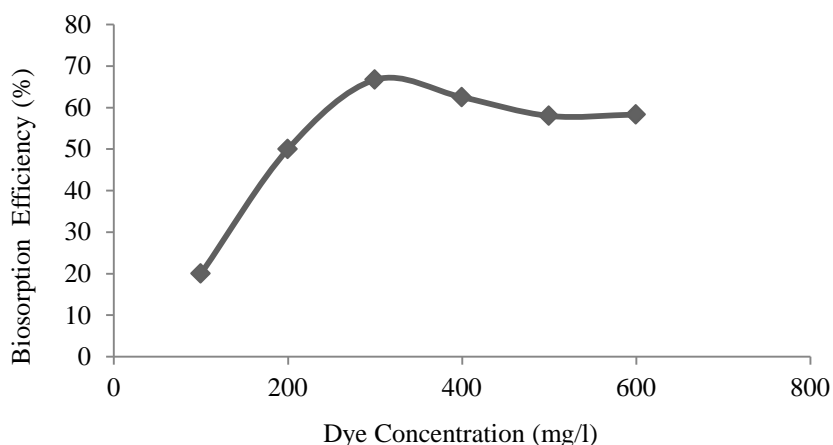
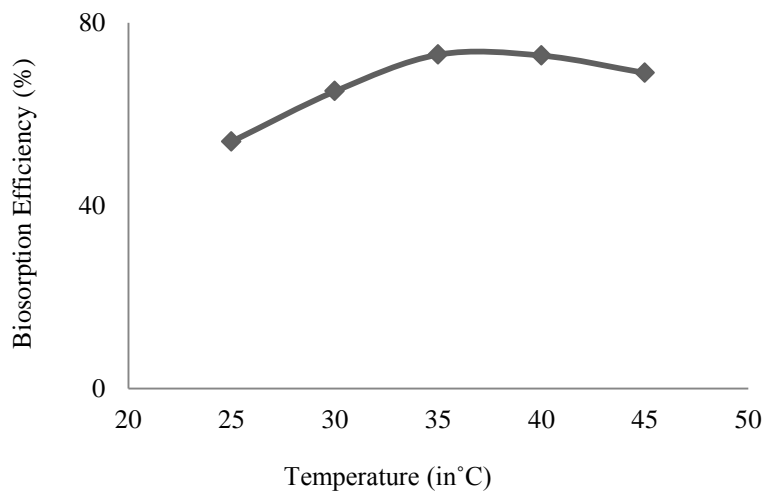
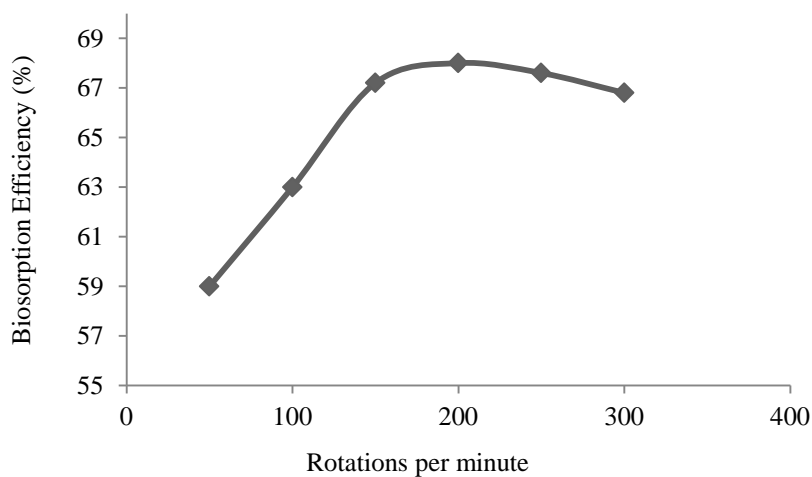


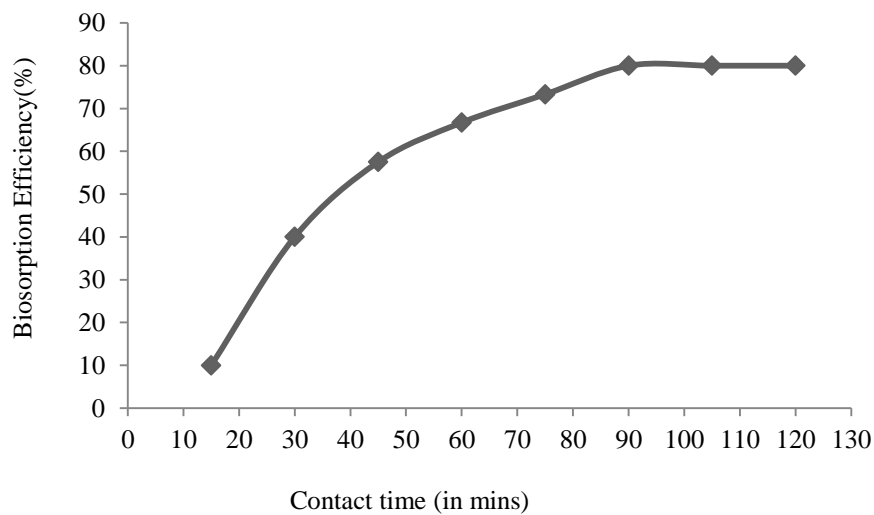
Figure-9: Effect of dye concentration on Biosorption efficiency.



**Figure-10:** Effect of Temperature on Biosorption Efficiency



**Figure-11:** Effect of rpm on biosorption efficiency.



**Figure-12:** Effect of contact time on biosorption efficiency.

The evolution of the biosorbed amount of dye with the contact time indicates that the equilibrium was attained in about 90 minutes for the adsorption of dye.

**Biosorption Kinetics: Pseudo-First Order Kinetics:** Pseudo First Order model describes the adsorption of solute onto Since it is the initial step of the optimization process the initial dye concentration was taken as 300mg/l with a solution pH of 8. The contact time was set to be 90 minutes with an rpm of 150 under 30°C in a temperature controlled incubator. The reaction was continuously monitored and the absorbance value is obtained at regular intervals of 15 minutes using a Colorimeter. The obtained values were tabulated and presented as a graph to understand the effect of the varied range of adsorption dosage on the biosorption and to calculate the biosorption efficiency of the adsorbent following first order mechanism<sup>2</sup>.

$$\frac{dq_1}{dt} = k_1 (q_e - q_t)$$

**Pseudo-Second Order Kinetics:** This model assumes that the rate of adsorption of solute is dependent on the amount of solute on the surface of the adsorbent. The driving force is proportional to the number of active sites available on the adsorbent<sup>2</sup>.

$$\text{Driving Force} = (q_e - q_t)$$

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$$

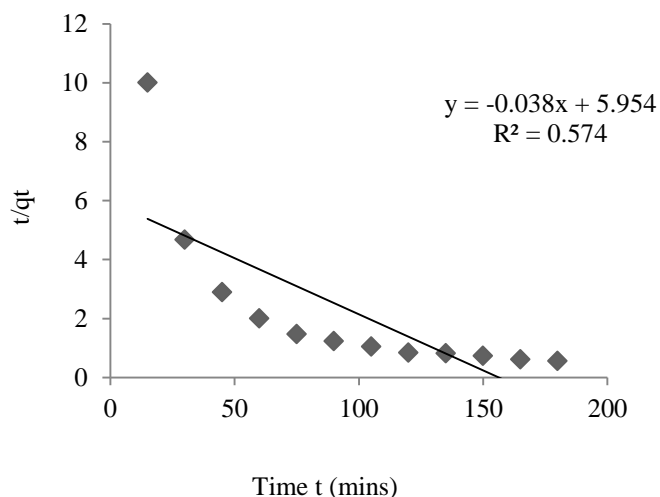
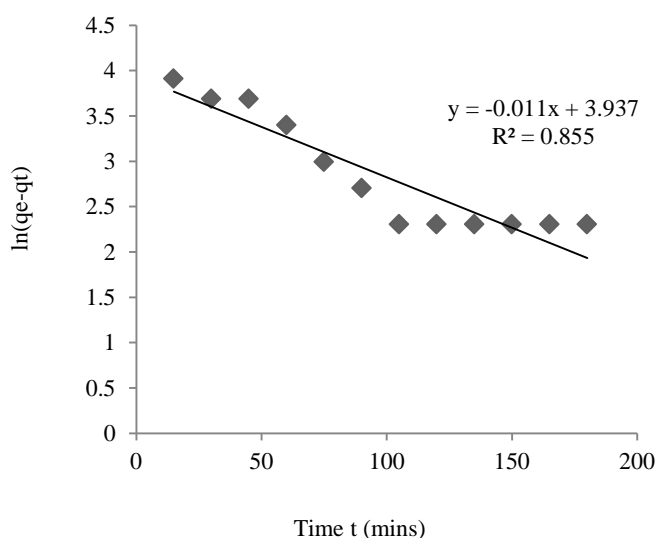
Kinetic data were analysed on the basis of the regression coefficient ( $r^2$ ) and the amount of dye removed per unit weight of

the biosorbent. The Pseudo first order kinetics was well observed than the Pseudo second order kinetics, since the initial number of sites available is higher and thus the rate of biosorption is greater at the initial stages.

## Conclusion

In this study, the raw and unmodified powder from the leaf sheath of the betel nut tree (*Areca catechu*) was used as an adsorbent for the removal of basic yellow dye from its synthetic aqueous solution. The Fourier Infrared spectrum of the leaf sheath powder of *Areca catechu* (biosorbent) before and after biosorption was analysed in the range of 600-3600 $\text{cm}^{-1}$ . The biosorption efficiency of the biosorbent was evaluated using various parameters which are necessary for an effective biosorption process. From which, it was found that with an adsorbent dosage of 350mg/l, pH 10, initial dye concentration of 300mg/l with a rpm of 200 provides effective biosorption efficiency of 80%. The biosorption process attained equilibrium at 90 minutes of contact time at 35°C temperature. The adsorption was well described by the Pseudo first order model. The Langmuir model gave the best fit for the experimental data than the Freundlich isotherm proving the monolayer adsorption of the dye on the surface of the biosorbent. Overall efficiency of the biosorption process was found to be 80%.

**Future Work:** i. Determining the biosorption ability of the leaf sheath powder after surface modification, ii. Desorption studies for the removal of basic yellow dye from the leaf sheath powder, iii. Interpreting the desorption efficiency with suitable isotherms and kinetics model, iv. Analysing the ability of the leaf sheath powder to act as an independent bio remediating agent.



**Figure-13 & 14:** Pseudo First Order kinetics and Pseudo Second Order Kinetics.

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