

## Research Article



# Lemon Juice and Microwave Assisted Modification of Date Seed Husk for Arsenic Biosorption

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**Abstract** | Arsenic poisoning and its removal from drinking water has become a serious issue now a days. For arsenic removal batch studies were conducted using low cost adsorbent (raw date seeds husk and lemon juice microwave activated date seeds husk) by taking 25ppm/50ml initial concentration of  $\text{NaAsO}_2$ . Best removal (90%) was achieved for Lemon juice microwave activated date seed husk LMDS (time= 30mins, agitation speed= 150 rpm, pH= 5, adsorbent dose= 0.1 g). While 85% removal efficiency was observed for Raw date seed husk RDS (time= 45mins, agitation speed=250 rpm, pH= 4 and adsorbent dose= 0.5 g). The adsorption isotherms (Freundlich and Langmuir) and adsorption dynamic kinetic studies were also conducted. The comparison between the FTIR, EDX and SEM of RDS and LMDS revealed that with lemon juice microwave activation of adsorbent there were activation of more active sites which in turn increased the removal efficiency.

**Received** | May 29, 2019; **Accepted** | December 23, 2019; **Published** | December 29, 2019

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**Citation** | Khan, T.M., Riaz, I., Hameed, S. and Khan, B., 2019. Lemon juice and microwave assisted modification of date seed husk for arsenic biosorption. *Journal of Innovative Sciences*, 5(2): 106-114.

**DOI** | <http://dx.doi.org/10.17582/journal.jis/2019/5.2.106.114>

**Keywords** | Biosorption, RDS, LMDS, Natural modification, SEM, EDX, Batch studies

## 1. Introduction

Arsenic toxicity is now recognized as a global health problem associated with almost more than 21 countries (Smith et al., 2000). In nature arsenic exists in the form of ores (powdery, amorphous and crystalline). Different anthropogenic and natural sources (mining and smelting processes, pesticide use and coal combustion) are responsible for water contamination by arsenic (Raj et al., 2013). Arsenic in environment (groundwater and in turn in drinking water) is not only creating the other health problems (respiratory, renal, neurological, cardiovascular, mutagenic etc.) but also has a prominent role in creating various types of cancer (Hall, 2002; Roy et al., 2013; Saha et al., 1999; Asif and Chen, 2017) in several developing regions (Brouwer et al., 2007). Ground water arsenic is present

in the form of As V (arsenate) and As III (arsenite) (Kumari et al., 2005; Wasiuddin et al., 2002; Amin et al., 2006; Roy et al., 2013).

For arsenic remediation various techniques involving adsorption were considered (Sharma and Bhattacharya, 2017). Various adsorbents used for arsenic removal are DE-4 resin (Qureshi and Sahabuddin, 2012), red mud (Bauxsol), a waste from aluminum manufacturing (Genc et al., 2003), iron coated brown seaweed (*Sargassum muticum*) (Vieira et al., 2017), Ceria ( $\text{CeO}_2$ ) coated powdered activated carbon (Sawana et al., 2017), modified fungal biomass of *Aspergillus niger* coated with iron oxide (Pokhrel and Viraraghavan, 2006), sand was coated with ferric chloride and used as filtering media (Devi et al., 2014), limestone-based material is effective for

reducing arsenic (Davis et al., 2018) and silica based chitosan beads (Malwal and Packirismy, 2017).

These conventional methods are reported to be noneconomical, less efficient and not safe environmentally (Eccles, 1999; Barakat, 2011; Azimi et al., 2016). A best and alternative method for arsenic remediation (Kratochvil and Volesky, 1998; Asif and Chen, 2017) is biosorption which utilizes natural, dead biomass (plants, agricultural wastes) or microorganisms (Roy et al., 2017; Ozer et al., 1998; Dimme et al., 2017; Amin et al., 2017; Sidhu et al., 2014; Javanbakht et al., 2014). These natural materials are excessively available having low or no cost and also have high decontamination ability. Biosorbents for removal of arsenic can be used in activated or in natural form (activated cashew shells, millet stalks, seed powder (Raj et al., 2013), *Azadirachta indica* bark powder (Roy et al., 2017), rice husk (Asif and Chen, 2017) and biochars derived from rice husk (Agraftioti et al., 2014).

The metal decontamination ability is attributed due to the presence (Basso et al., 2002; Chen et al., 2010), activation and modification of various groups ( $-\text{COOH}$ ,  $\text{OH}$ ,  $\text{NH}_2$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$ ) (Deng et al., 2003). These groups when introduced or activated can enhance the adsorption power of adsorbent (Oliveira et al., 2009). Metal entrapment by these natural residues is due to metal substrate interactions by various processes (Srivastava and Anil, 2016). So far biosorbents can be activated by using different chemicals like  $\text{FeCl}_3$ ,  $\text{ZnCl}_2$  (Liu et al., 2016),  $\text{H}_2\text{O}_2$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{NaOH}$  (Shwantes et al., 2016), urea (Rehman et al., 2013) and thiourea (Salman et al., 2014).

Various biosorbents were successfully being used for removing heavy metals due to their different up take capacities ( $q_{\text{max}}$  values) Table 1. Present study was designed to check the effectiveness of *Phoenix dactylifera* seeds (date seeds) belonging to family *Arecaceae*, an agricultural waste in removing arsenic ions from aqueous solution. There are many areas in Sindh and Baluchistan which are producing high quality dates of different varieties. Their seeds can be reused in different ways. In present study two types of biosorbents i.e., raw date seeds husk (RDS) and microwave assisted lemon juice modified date seed husk (LMDS) were used for the first time as a new biosorbent and its modification with lemon juice is totally ecofriendly instead of using various chemicals as modifiers or activators.

**Table 1: Comparison of sorption capacities of different biosorbents for the removal of Heavy metals.**

Biosorbent	Heavy metal	$q_{\text{max}}$ mg/g	Reference
Oil palm shell	Pb (II)	3.39	(Chong et al., 2013)
Oil palm shell	Cu (II)	1.75	(Chong et al., 2013)
Palm shell	Hg (II)	83.33	(Ismail et al., 2013)
Corn straw	Cd (II)	38.91	(Chi et al., 2017)
Corn straw	Pb (II)	28.99	(Chi et al., 2017)
Raw date seeds	As (III)	1.3332	Present study
Lemon juice microwave activated date seeds	As (III)	1.4828	Present Study

By adding lemon juice there is the introduction of additional  $-\text{COOH}$  groups on adsorbent surface (date seed husk) and with microwave radiation these groups are much more activated as  $\text{COO}^-$  thus providing more adsorbent sites for arsenic ions removal.

The capability of RDS and LMDS for arsenic ions removal was checked by studying isothermal, kinetic and batch studies.

## 2. Materials and Methods

Shaker (yellow line), Atomic Absorption spectrometer, PH meter, Microwave oven (2,450 MHz), Midac FTIR 2000 spectrometer ( $406\text{--}7800\text{cm}^{-1}$ ), EDX, SEM, 0.1M  $\text{NaOH}$ , 0.1M  $\text{HCL}$  (MERCK), Sodium Arsenite salt ( $\text{NaAsO}_2$ ) and lemon juice.

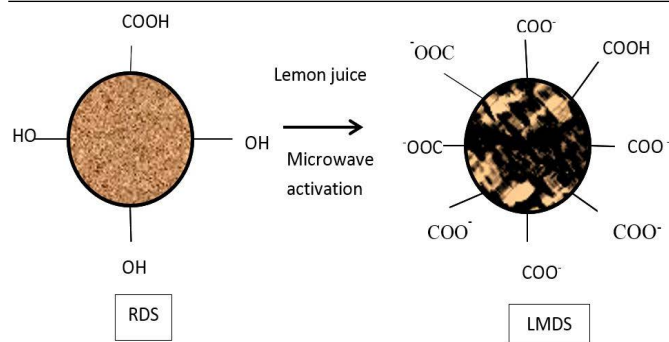
### 2.1 Preparation of solution

Stock solution of 1M Sodium Arsenite salt ( $\text{NaAsO}_2$ ) was prepared. From this stock solution 25 ppm/50 ml volume of solution was prepared and mixed into the activated as well as non-activated date seed husk.

### 2.2 Preparation of biosorbent

**Preparation of raw date seed husk:** Date seeds (*Phoenix Dactylifera*) an easily available raw material were purchased from seed bank of Lahore. These were then washed, dried, grinded and sieved (60 ASTM). This was raw date seed husk (RDS).

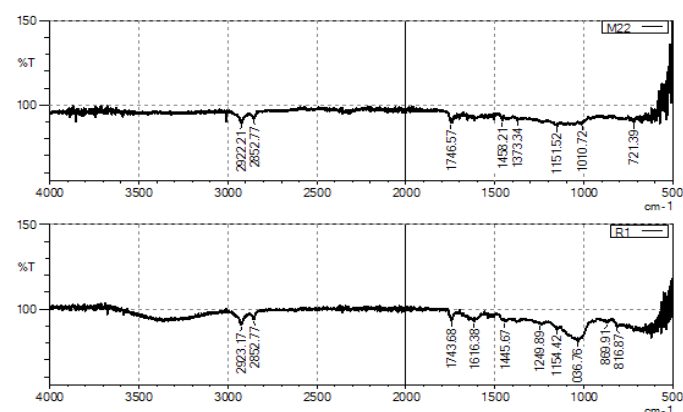
**Preparation of modified Date Seed husk:** 60g of the preserved raw date seed husk (RDS) and lemon juice (1:1) was mixed fairly. This was then subjected to microwave oven for 25 min. to get lemon juice microwave activated date seed husk (LMDS) Scheme 1.



**Scheme 1: Surface modification of RDS with lemon juice and microwave radiation.**

### 3. Results and Discussion

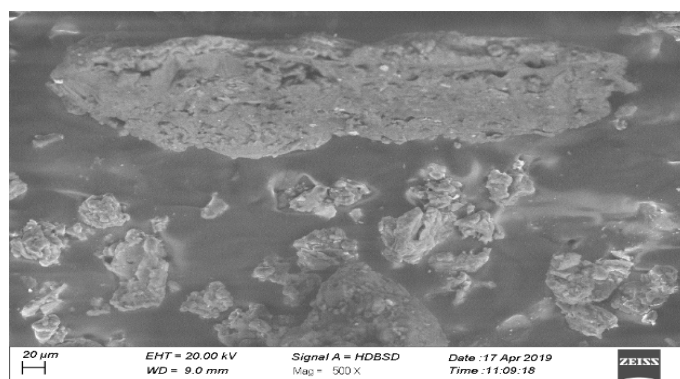
**FTIR Analysis:** FTIR spectra of raw date seed husk (RDS) and lemon juice microwave activated date seed husk (LMDS) [Figure 1](#) showing various functional groups on the surface of RDS and LMDS. LMDS showed the presence of additional peaks at  $1746.57\text{ cm}^{-1}$  (C=O Stretching),  $1458.21\text{ cm}^{-1}$  (C-H Stretching),  $1373.34\text{ cm}^{-1}$  (O-H Bending),  $1151.52\text{ cm}^{-1}$  (C-O Stretching),  $1010.72\text{ cm}^{-1}$  (C-N Stretching) and  $721.39\text{ cm}^{-1}$  (C=C Bending). RDS showed different peaks in the region of  $1616.38\text{ cm}^{-1}$  (C=C Stretching),  $1445.67\text{ cm}^{-1}$  (C-H Bending),  $1249.89\text{ cm}^{-1}$  (C-O Stretching),  $1154.42\text{ cm}^{-1}$  (C-O Stretching),  $1036.76\text{ cm}^{-1}$  (S=O Stretching),  $869.91\text{ cm}^{-1}$  (C=C Bending),  $816.87\text{ cm}^{-1}$  (C=C Bending). There was also shift in peaks of RDS from  $2923.17\text{ cm}^{-1}$  to  $2922.21\text{ cm}^{-1}$ ,  $1743.68\text{ cm}^{-1}$  to  $1746.57\text{ cm}^{-1}$ ,  $1445.67\text{ cm}^{-1}$  to  $1458.21\text{ cm}^{-1}$ ,  $1154.42\text{ cm}^{-1}$  to  $1151.52\text{ cm}^{-1}$  and  $1036.76\text{ cm}^{-1}$  to  $1010.72\text{ cm}^{-1}$  after modification with lemon juice and microwave activation. Thus the presence of these groups were involved in biosorption of As (III) by *Phoenix Dactylifera*. LMDS had more active sites and had more functional groups indicating the involvement of these groups in biosorption of As (III).



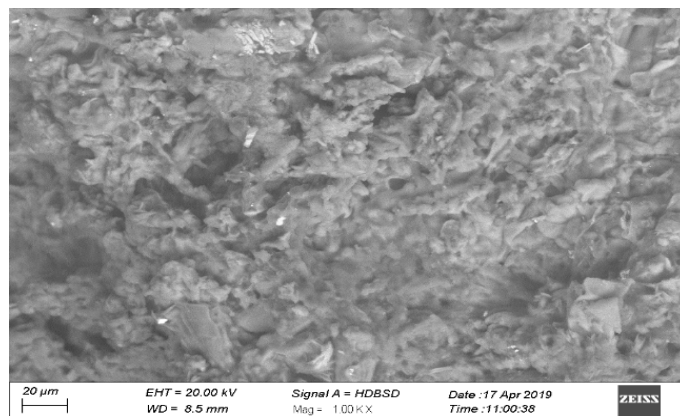
**Figure 1: Overlay FTIR spectra of RDS and**

### LMDS.

**SEM Analysis:** It was noticed that the surface of RDS was rough and consisted of particles of various shapes and sizes. Particles were large in size providing small surface area [Figure 2a](#). After lemon juice microwave activation surface of LMDS had become irregular and wavy and had holes which were unevenly spread on the small size particles of LMDS [Figure 2b](#). After activation of sample with lemon juice surface morphology was changed and increases in surface area consequently giving higher sorption capacity of the sorbent.



**Figure 2a: SEM image of RDS.**



**Figure 2b: SEM image of LMDS.**

**EDX Analysis:** EDX image of RDS [Figure 3a](#) shows its elemental composition as having Ca, K, Na and no arsenic but for LMDS an additional prominent peak is observed due to retention of arsenic on the surface of biosorbent [Figure 3b](#).

#### 3.1 Batch studies

**Effect of shaking speed:** It was observed that adsorption of Sodium Arsenite ( $\text{NaAsO}_2$ ) (25ppm/50ml) had increased with the increased shaking speed (50 – 400 rpm) for both RDS and LMDS. At 250 rpm for RDS adsorption reaches a maximum of 60.50 % and at 150 rpm for LMDS maximum



adsorption was 90.25 % [Figure 4a](#). Moderate speed provided more active sites as compared to low speed and further increase in speed decreased the contact time resulting in desorption.

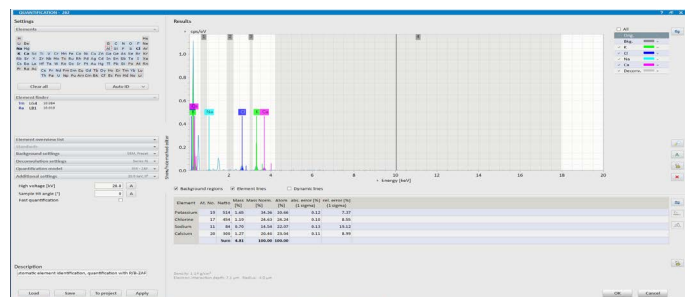


Figure 3a: EDX image of LMDS without arsenic.

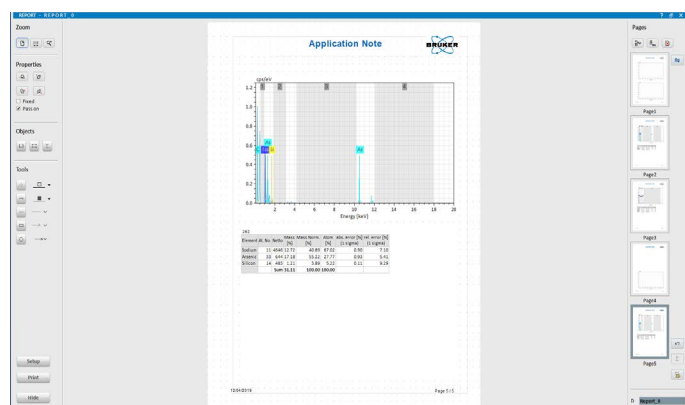


Figure 3b: EDX image of LMDS with arsenic.

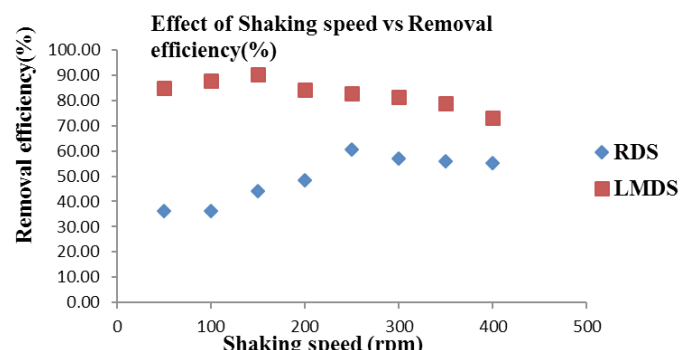


Figure 4a: Effect of shaking speed for Arsenic removal onto RDS and LMDS. Adsorbent dose= 0.3 g, Solution concentration (25 ppm / 50 mL), Contact time= 30 minutes.

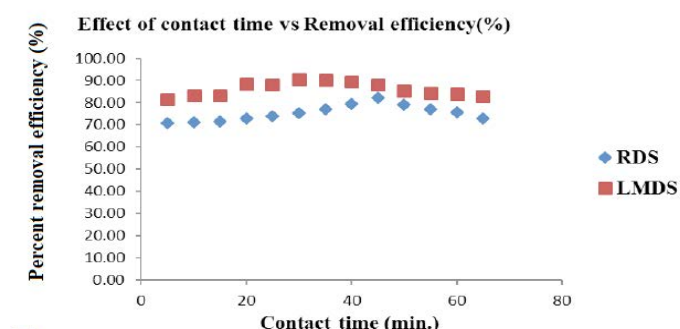


Figure 4b: Effect of contact time for Arsenic

removal onto RDS and LMDS. Adsorbent dose= 0.3 g, Shaking speed (RDS, LMDS= 250 rpm, 150 rpm), Solution concentration (25 ppm / 50 mL).

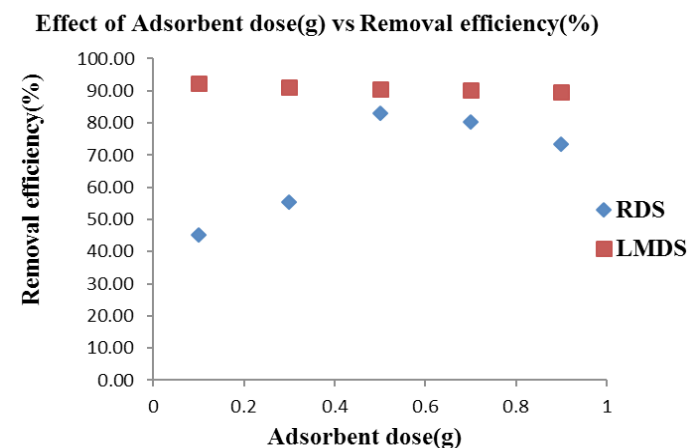


Figure 4c: Effect of adsorbent dose (g) on Arsenic removal onto RDS and LMDS. Shaking speed (RDS, LMDS= 250rpm, 150 rpm), Solution concentration (25 ppm / 50 mL), Time= 30 minutes.

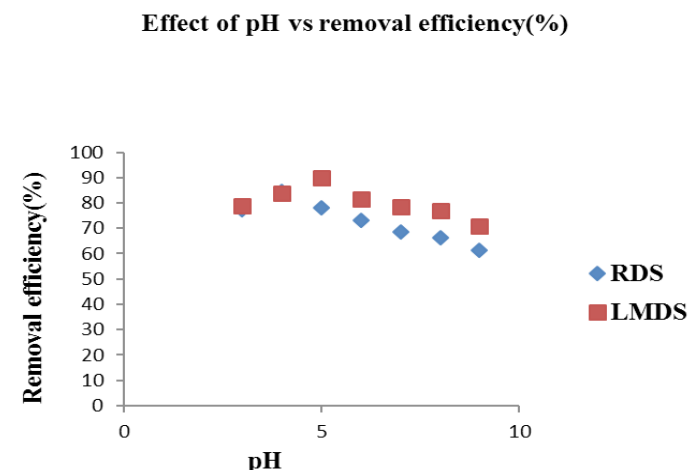
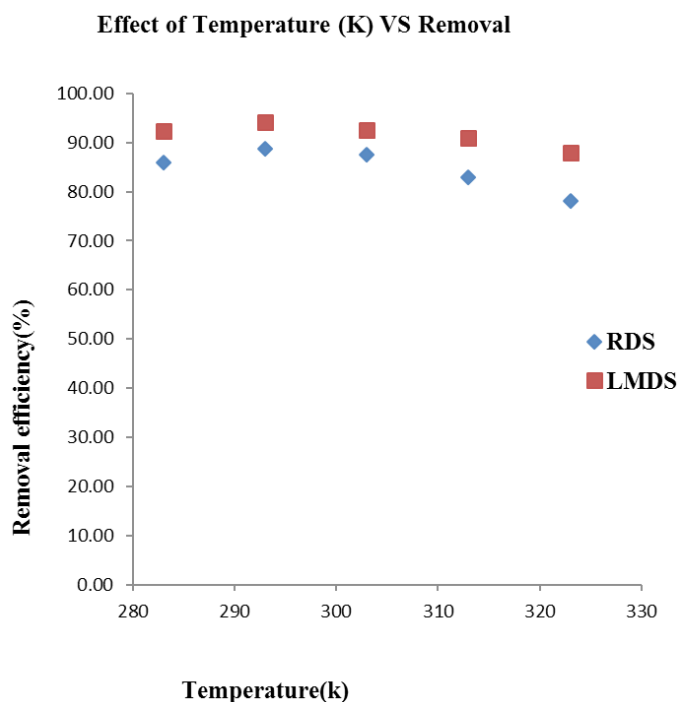


Figure 4d: Effect of pH on lead removal onto RDS and LMDS. Adsorbent dose (RDS, LMDS= 0.3g), Shaking speed (RDS, LMDS= 250 rpm, 150 rpm), Solution concentration (25 ppm / 50m L), Time=30 minutes.

**Effect of contact time:** Removing capacity (%) of RDS and LMDS was increased with the increase in their time of contact [Figure 4b](#). Maximum removal efficiency (%) of RDS was 82.25 % at 45 minutes contact time whereas LMDS had 90.59 % removal efficiency at 30 minutes contact time. Therefore, the 30 minutes contact time was chosen for further batch adsorption experiment.

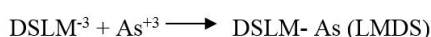
**Effect of adsorbent dose:** Adsorption behaviour of RDS and LMDS was studied by keeping the adsorbent amount from 0.1g–0.9g [Figure 4c](#).

Maximum adsorption occurred when RDS is 0.5 g and LMDS was only 0.1 g increase in adsorption was due to the increased sites and then decrease was due to their aggregation and decreased surface area.



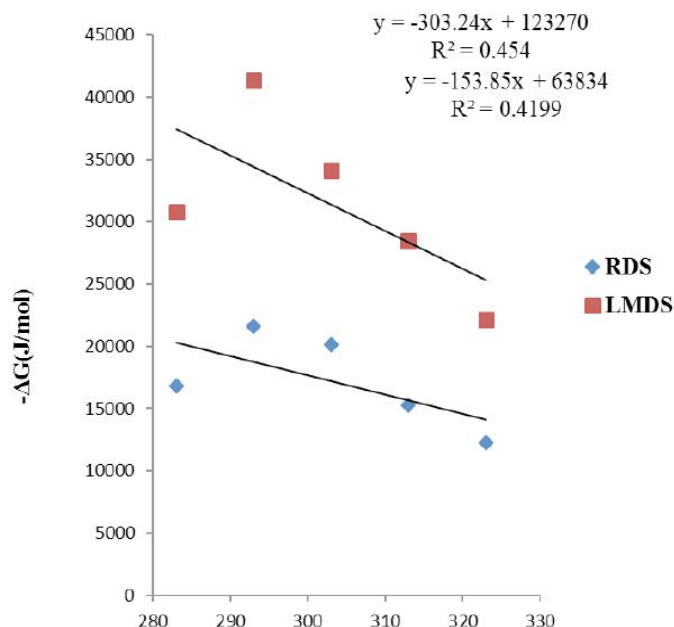
**Figure 4e: Effect of temperature (K) on Arsenic removal onto RDS LMDS. Adsorbent dose = 0.3g, Shaking speed (RDS, LMDS = 250 rpm, 150 rpm), Solution concentration (25 ppm / 50m L), Time=30 minutes.**

**Effect of pH:** The pH (3-9) also effected the adsorption capacity of RDS and LMDS due to the presence of different forms of carboxylic group on biosorbent surface at different pH.  $As^{+3}$  adsorption was low at pH 3 because at pH 3 the dominating specie was  $-COOH$  and it then increases and reaches the highest at pH 5 where  $-COO^-$  was mainly present on adsorbent surface. For RDS  $As^{+3}$  maximum removal efficiency (%) was at pH 4 (84.625 %) and for LMDS at pH 5 (89.75 %) Figure 4d. At still higher pH (6.0) adsorption decreases due to the precipitation of  $As^{+3}$  as insoluble metal hydroxides. The mechanism of  $As^{+3}$  biosorption is:



**Temperature dependence:** Temperature is an important parameter to study extent of adsorption. Experiment were conducted in the temperature range 283.0 K - 333.0 K in which 293.0 K was the

suitable temperature for both the sorption of  $As^{+3}$  using RDS (90.16 %) and LMDS 93.53 % Figure 4e. Above 293.0 K adsorption decreases due to very high movement of  $As^{+3}$  and decrease in interaction between adsorbent surface and adsorbate. The value of  $\Delta G$  for RDS and LMDS showed that the reaction is spontaneous and its greater value for LMDS than RDS means that adsorption was more favorable with LMDS Figure 5.



**Figure 5: Thermodynamic study of RDS, LMDS.**

### 3.2 Isothermal study

**Langmuir Isotherm:**  $q_{max}$  values for RDS and LMDS were 1.3332 mg  $g^{-1}$  and 1.4828 mg  $g^{-1}$  Table 2. Greater  $q_{max}$  for LMDS had more active sites as compared with RDS.  $R_L$  values of LMDS and RDS were 0.0237 and 0.0335 respectively which indicated that data is not according Langmuir Figure 6a.

**Table 2: Isothermal parameters for Arsenic sorption onto LMDS and RDS.**

Adsorbent	Langmuir	Freundlich
LMDS	$q_{max}(mg/g)$	1.4828
	$b(L/g)$	1.6503
	$R^2$	0.7581
	$R_L$	0.0237
RDS	$q_{max}(mg/g)$	1.3332
	$b(L/g)$	1.1527
	$R^2$	0.7855
	$R_L$	0.0335

**Freundlich isotherm:** Magnitude of  $K_F$  and  $n$  (Freundlich constant) indicated high adsorptive

capability of *Phoenix Dactylifera*. The value of  $K_F$  for LMDS and RDS were 2.7064 and 2.605 respectively Table 2. The value of  $R^2$  for LMDS and RDS were 0.9245 and 0.9226 respectively which is a good fit of Freundlich isotherm Figure 6b.

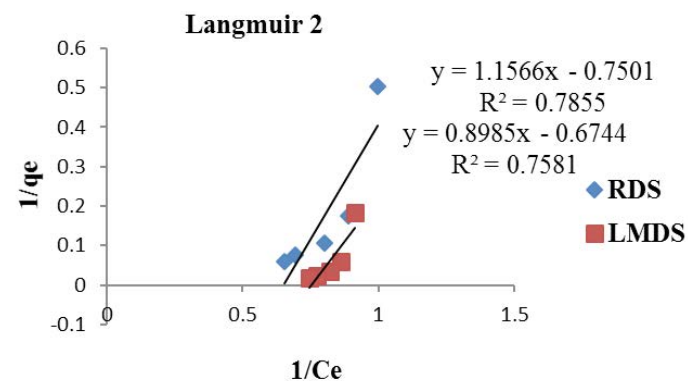


Figure 6a: Langmuir 2 Isotherm for RDS, LMDS.

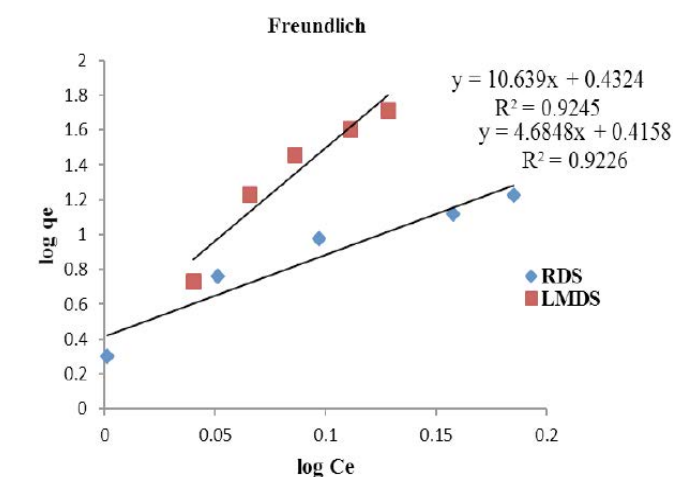


Figure 6b: Freundlich Isotherm for RDS, LMDS.

Table 3: Kinetic Study for Arsenic sorption onto LMDS and RDS.

Pseudo first order kinetic Model				
Adsorbent	$q_e$ (exp)	$K_1$	$q_e$ (cal)	$R^2$
RDS	11.79	0.00007	2.582	0.0425
LMDS	12.4896	0.0002	2.6072	0.3419
Pseudo second-order kinetic model				
Adsorbent	$q_e$ (exp)	$K_2$	$q_e$ (cal)	$R^2$
RDS	1.92864	0.2151	0.216	0.9936
LMDS	3.6049	0.2151	0.4381	0.9966

**Kinetics studies:** *Phoenix Dactylifera* kinetics studies were not according to pseudo first order reaction but is according to second order Table 3 as the experimental values of  $K_1$  and  $q_e$  were not according to calculated values and the  $R^2$  (correlation coefficients) were low Figure 7a. Whereas  $k_2$  (pseudo second order rate

constant) and  $q_e$  values agreed with experimental data and  $R^2$  values is a good fit for pseudo second order reaction Figure 7b.

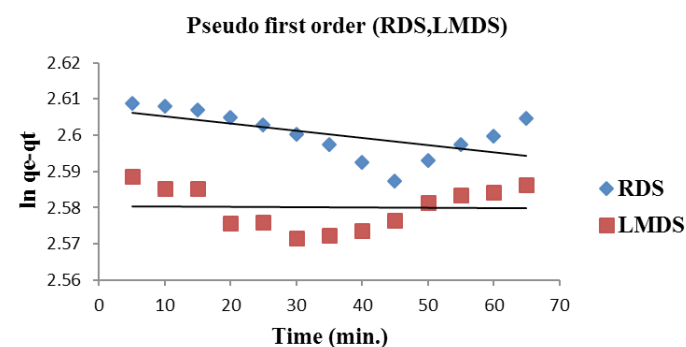


Figure 7a: Pseudo first order kinetics for (RDS, LMDS).

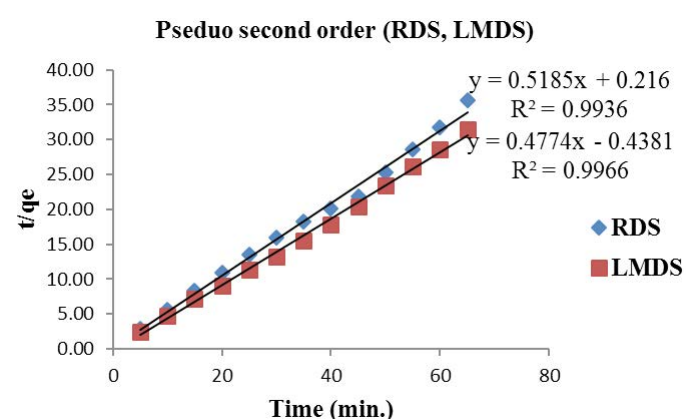


Figure 7b: Pseudo second-order kinetics for (RDS, LMDS).

## Conclusions and Recommendations

On the basis of the result it can be concluded that Date seed (*Phoenix Dactylifera*) could be successfully used for As (III) removal from aqueous solutions. The results obtained suggest that LMDS is a good adsorbent as compared to RDS because it had more active sites. Experimental data fitted very well to the Freundlich adsorption isotherm models. FTIR spectra identified different functional group present in the *Phoenix Dactylifera*. Process adopted is simple and economically valueable. It could be used for sorption of different other heavy metals like Cd, Pb etc. and also for different dyes such as methylene blue etc. from water. Activation by lemon juice was very effective, low cost and at the end of reaction sludge or by products were not formed. This method is easily applicable in agricultural country where a lot of agricultural waste is present and lemon juice is also abundantly available. This method can be applied on industrial scale to remove contaminants from waste water.

## Acknowledgement

All this research work was conducted and supported by the Central Lab. of Lahore College for Women University, Jail Road Lahore Pakistan.

## Authors Contribution

All this work was planned and supervised throughout by Dr. Tahira Moeen Khan. Iram Riaz and Shahida Hameed helped in sample preparation and statistical data. Prof. Dr. Bushra Khan helped in the analyzing of data.

## Conflict of interest

The authors have declared no conflict of interest.

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