

Removal of Organic Matters, Phosphates and Nitrates Present in Dairy Industrial Wastewater Using Jujube Seeds Biocoagulant

Nouria Nabbou¹

¹Architecture and Environmental Heritage Laboratory (ARCHIPEL), Faculty of Technology, Department of engineering process, Mohammed Tahri University of Bechar, (08000) Algeria nabou.nouria@univ-bechar.dz; ORCID identifier: <https://orcid.org/0000-0002-5725-5077>

Abstract

This study aims to remove of organic matter, nitrate and phosphate present in industrial wastewater from a dairy facility in Bechar Algeria using the coagulation–sedimentation process. A natural coagulant based on jujube seed powder (*Zizyphus lotus*) was tested. Jar Test experiments were conducted to study the effect of various doses on pH, conductivity, COD, TSS, nitrates, and phosphates.

The results showed that the natural coagulant is effective, achieving removal efficiencies of: COD (90%), TSS (87.5%), NO_3^- (98.5%), PO_4^{3-} (70%) and OM (33.2%) at an optimal dose of 2.5 g/L without pH adjustment. Thus, jujube seed powder is a promising eco-friendly and cost-effective alternative for industrial wastewater treatment.

Keywords: organic matter, nitrates, phosphates, coagulation–sedimentation, biocoagulant, jujube seeds, dairy industry

I. INTRODUCTION

Water is a daily partner of humans used to meet their daily consumption and hygiene needs; it is used for drinking, cooking food, food production and product processing, personal hygiene and sanitation of living conditions, electricity production, and recreational use [1].

Population growth and economic and urban development lead to an increase in the amount of water consumed. These results in a significant increase in the volumes of liquid discharges and the flows of polluting organic matter they generate. The production of wastewater, often discharged into the natural environment without prior treatment, poses a considerable threat to ecosystems and generates numerous waterborne diseases that can lead to certain epidemics. the search for adequate purification methods and the reuse of treated wastewater effluents has become an attractive option and an essential alternative [2]. Several conventional technologies such as adsorption, membrane processes, flotation, coagulation, flocculation, biological or electrochemical methods have been successfully applied in water treatment [3,4]. Most conventional processes require the introduction of chemicals or additives (acids, bases, chlorines, etc.). The addition of these substances is quite complex and requires thorough dosage tests because it also leads to the generation of a large volume of dangerous and toxic sludge that requires additional treatment. Other processes generate harmful by-products for humans, animals, and aquatic life (Disinfection By-products "DBPs"; case of chlorination) [5,6]. Coagulation and flocculation are key processes in water purification and particularly in wastewater treatment, due to their efficiency, simplicity, and low energy requirements. They can be applied alone or in conjunction with other techniques [7,8]. Classically, mineral coagulants and synthetic flocculants are widely used for this purpose, due to their extraordinary ability to eliminate colloidal matter. Nevertheless, these are mineral additives comprising metallic salts such as aluminum polychloride and synthetic polymers such as polyacrylamide [8,9]. Although these types of chemical coagulants are widely applied, they are somewhat costly and produce a large volume of non-biodegradable sludge requiring special handling, they significantly affect the pH of the treated water and generate toxic by-products that are harmful to human health [10,11]. The residual amount of aluminum in treated water can cause Alzheimer's disease [12]. In response to global concerns about reducing the harmful effects of chemical coagulants, the development of wastewater treatment agents with specific customized characteristics, based on renewable and natural resources, is becoming the focus of future strategies for more sustainable wastewater treatment [13]. Numerous studies have concluded that biomaterials, extracted from microorganisms of animals or plants, can be of great interest because they are low-cost natural products, characterized by their environmentally friendly behavior, biodegradability, non-toxic nature, and abundance. The sludge produced by these biomaterials is not only harmless but also four to five times less voluminous than the chemical sludge produced by alum [14,15]. The main objective of our work is to study the possibility of using jujube seed powder as an alternative

coagulant (biocoagulant) to organic matter, nitrate, and phosphate removal present in liquid effluent from a dairy industry located in Bechar. Our choice was directed towards the valorization of a plant waste, which is jujube pits, to treat a liquid effluent from a dairy industry that is discharged untreated. The jujube plant is a very abundant local species that has not yet been massively exploited and presents promising virtues

II. MATERIALS AND METHODS:

III. III.1 Effluent Sampling

As part of our study on the evaluation of quality, samples were collected at the wastewater discharge point of the dairy (Figure 1), located in the industrial zone of Ouakda, Bechar city. The water sample was collected according to the protocol described by Rodier et al., [16] using sterile glass bottles, ensuring that the sample was as representative as possible.



Figure 1. Collection basin for dairy effluents [4].

III.2 The plant material (the bio-coagulant)

The seeds of *Zizyphus lotus* (jujube) were collected in the Bechar region in the month of September. The jujube seeds are washed with running water to remove all kinds of dust or adhering impurities as well as water-soluble substances until clear wash water is obtained, then rinsed with distilled water and dried at 110 °C for 24 hours. They were ground using a grinder, then the obtained powder was sieved using a 400 μ m mesh sieve (Figure 2).



Figure 2: steps for preparing jujube seeds powder

II.2.1 Jujube seeds characterization

a) Characterization by Fourier transformed infrared spectroscopy (FTIR)

Infrared spectroscopy (IR) is an important technique used for the identification of characteristic functional groups on the surface of the adsorbent. These groups are often responsible for the adsorbent-adsorbate bonds. It involves measuring the absorption of different IR frequencies by a sample traversed by an IR beam. The infrared range extends from 0.78 μ m to 1000 μ m. It is arbitrarily divided into 3 categories: near infrared (0.78 to 2.5 μ m or 12500-4000 cm^{-1}), mid infrared (2.5 to 50 μ m or 4000-400 cm^{-1}), and far infrared (50 to 1000 μ m or 400-10 cm^{-1}) [17]. Infrared analysis was performed using a

Fourier Transform Infrared Spectrometer FTIR-8300 (SHIMADZU) with a frequency range between 400 and 4000 cm^{-1} .

b) Determination of Ash Content

It allows us to know the proportion of organic matter that is part of the composition of the jujube pit and should be as low as possible. To determine the ash content, a mass of the sample is placed in a crucible, heated in a muffle furnace at 725°C for 1 hour and 30 minutes (Fig.13). It is then cooled in a desiccator and weighed.

Organic matter rate = $100 \times P_f / P_i$

P i: mass in grams of the crucible and the dry matter before incineration.

P f: mass in g of crucible with ashes [17].

c) Determination of pH_{PZC}

The pH_{PZC} or point of zero charge pH corresponds to the pH value at which the net charge of the solid surface is zero. This parameter is very important in adsorption phenomena, especially when electrostatic forces are involved in the mechanisms.

to determine the pH_{PZC} of jujube seeds. to determine this parameter, using the pH variation method which involves placing 50 cm^3 of NaCl solution (0.01 M) in closed flasks and adjusting the pH of each between 2 and 12 by adding NaOH or HCl solution (0.1 N). Next, 0.15 g of the material sample to be characterized is added to each flask. The suspensions must be kept agitated at room temperature for 48 hours. the final pH is then determined. The pH_{PZC} is the point where the final pH curve as a function of the initial pH intercepts the line final pH = initial pH. [18]. The surface is positively charged for pH values below the pH_{PZC} . Whereas, the surface is negatively charged for pH values above the pH_{PZC} .

III.3 Experimental setup and sample preparation

III.3.1 Preparation of coagulants in solution

Masses of the biomaterial powder (from 0.5 to 10g) were prepared in distilled water at different concentrations by stirring for 2 hours.

III.3.2 Experimental Protocol

The Jar-Test is the oldest, least expensive, simplest method, and therefore the most widely used. For more than fifty years, the Jar-Test has been the standard technique used to optimize the addition of coagulants and flocculants in the field of water treatment. It involves filling six beakers with the water to be treated and adding a gradually increasing dose of coagulant to each one, to reach the optimum doses necessary to obtain the clearest possible water. The Jar-Test used is of the VELP brand, featuring six stirrers with a rotation speed that can vary between 0 and 300 rpm. The blades are of the helical type and the volume of the beakers is 1 liter (Figure 3).

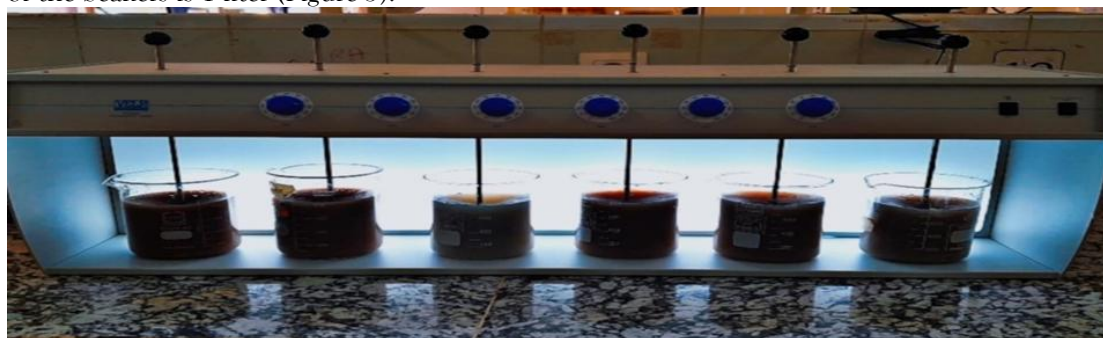


Figure 3: Jar test used for coagulation-sedimentation trials.

The first step; coagulation, is implemented by adding different amounts of the bio coagulant to the water samples, and the mixture is subjected to rapid agitation at 150 rpm for 20 minutes to ensure proper distribution of the material. For the second step; flocculation, a slow agitation of 120 rpm is carried out for 10 minutes to enlarge and consolidate the mini flocs formed. And finally, the third step is a natural sedimentation for 2 hours to separate the formed flocs from the more or less clear supernatant. The determination of physicochemical parameters (pH, EC, COD, OM, PO_4 , NO_3 , TSS) before and after treatment was carried out using the standard methods cited by Rodier et al., 2009 [19].

IV. RESULTS AND DISCUSSIONS:

IV.1 Biocoagulant characterization

IV.1.1 characterization of jujube powder

The infrared spectrum of the jujube seeds shown indicates that the surface of this material possesses very important characteristic functional groups, which can largely be responsible for the surface phenomena involved (Figure 4).

The analysis was conducted over a wavelength range of 4000-400 cm^{-1} . two broad bands, are identified, ranging between 3200 and 3400 cm^{-1} , which can be attributed to the stretching vibrations of hydrogen in the asymmetric O-H hydroxyl groups present in proteins, fatty acids, cellulose, carbohydrates found in jujube seeds, and the vibration of free hydroxyl groups (OH) in alcohols and phenols or adsorbed water [17].

Furthermore, a band at 2920 was detected, likely related to the asymmetric C-H stretching vibration of aliphatic molecules [20,21], present in alkyl groups, such as methyl and methylene groups [22, 23].

In the region between 1735 and 1420 cm^{-1} , several bands are observed and can be attributed to the stretching of the carbonyl C=O bond. The band observed at 1420 cm^{-1} corresponds to aromatic C-C [23]. Due to the heterogeneous nature of jujube pits, the carbonyl group can be linked to different parts, such as the lipid part of fatty acids at 1735.1 cm^{-1} or the protein part of amides at 1653.1 cm^{-1} [22].

Between 1500-1600 cm^{-1} , the presence of absorption bands corresponding to the valence vibration of C=C bonds in aromatics [24]. The bands observed in the range 1210-1320 cm^{-1} can be attributed to the vibration of C O bonds in the alcohols of esters and ethers.

An intense band observed at 1028.7 attributed to the asymmetric and symmetric stretching vibration of C-O-C. The main characteristic constituents of jujube seeds are: cyclic peptide alkaloids (e.g., jubanines A, amphibines -H); spinosine glycoside flavonoids (e.g., sinapoylspinosine), saponins (e.g., jujubosides A), phospholipids (e.g., phosphatidylglycerol), oleic acid [25,26].

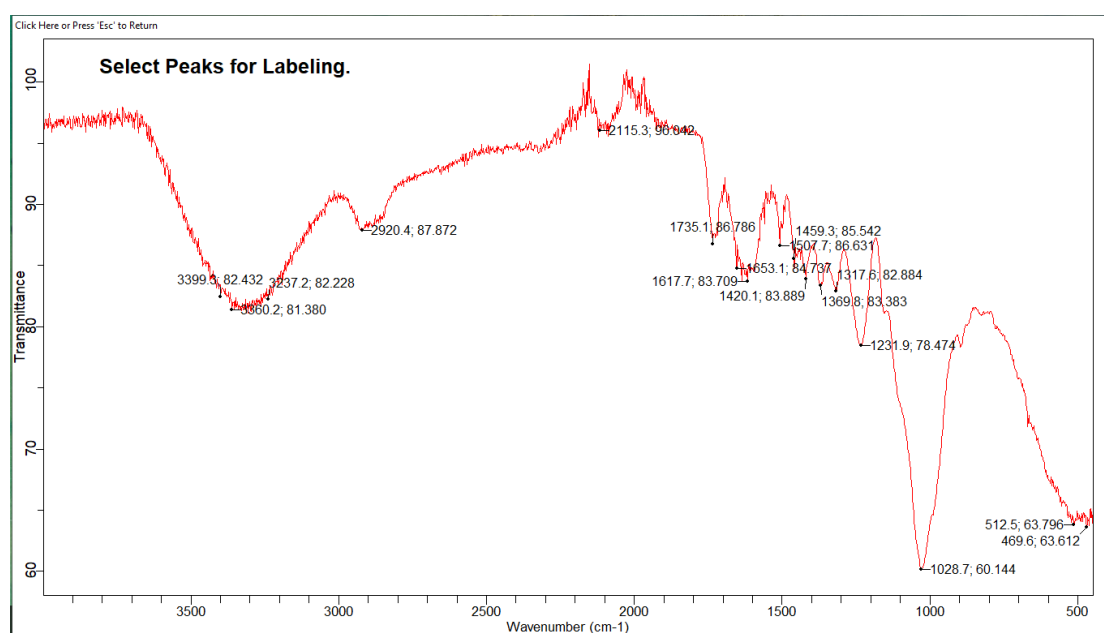


Figure 4: FTIR results of jujube seeds

IV.1.1 pH of the zero-charge point (pH_{PZC})

The pH_{PZC} corresponds to the pH value at which the net charge of the coal surface is zero. The results are presented graphically in (Figure 5). The surface of the material can acquire a positive or negative charge depending on the pH of the medium. When the pH of the solution is below the isoelectric point (pH_{PZC}) of jujube seeds ($\text{pH} < \text{pH}_{\text{PZC}}$), it exhibits a basic behavior that promotes the adsorption of anionic species [27]. On the other hand, if the pH of the medium is higher than the pH_{PZC} of the jujube seeds ($\text{pH} > \text{pH}_{\text{PZC}}$), their surface exhibits a negative charge, which promotes the adsorption of cationic species. The pH_{PZC} values of jujube seeds are equal to 7,5.

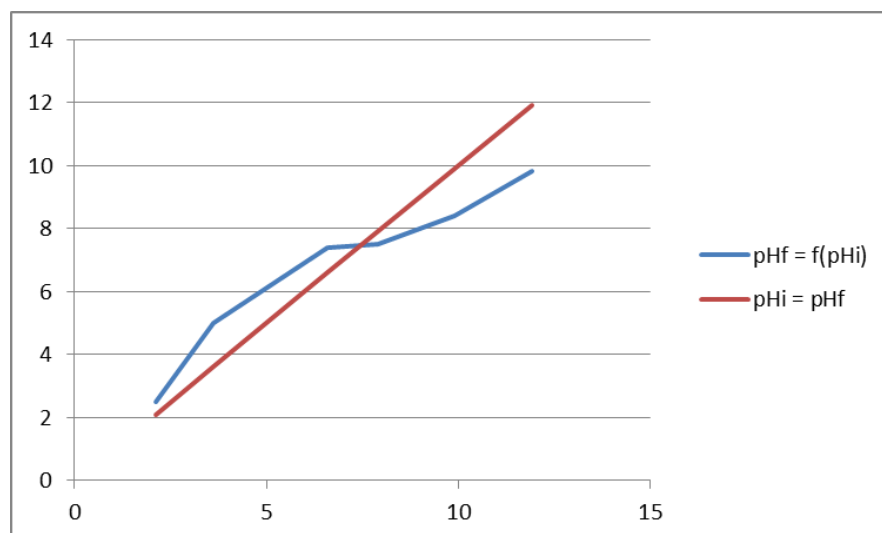


Figure 5: pH_{PZC} results of jujube seeds

IV.1.2 The ash content

The jujube seeds have very low ash content (1.09%), which is due to the presence of a small amount of minerals in the seeds. These results indicate that the jujube seeds used are characterized by the presence of a significant amount of organic matter, which constitutes the major component in their structure. The same ash content value was obtained by Mameri, 2017 for jujube seeds from the Bouira region (Algeria) [28].

IV.2 Results of liquid effluent treatment in the dairy industry

IV.2.1 Results of the physicochemical analyses of the liquid effluent from the dairy industry

The physicochemical characteristics of effluents are illustrated in Table 01.

Table 01: The physicochemical characteristics of liquid effluent from the dairy industry

characteristic	effluent	Algerian Standards (JORA, 2006) [29]
PH	6,1	6.5 – 8.5
DCO (mg/l)	4386,5	120
MO (mg/l)	190,4	5
MES (mg/l)	72	30
CE (ms/cm)	25	-
Nitrate (mg/l)	330,5	30
Phosphate (mg/l)	2,9	2

Generally speaking, the effluent water from the dairy industry is characterized by a pH relatively close to acidity and a high salt content of around 25 mS/cm. The phosphate level is 2.9 mg/l. The organic and nitrogen pollution generated in this discharge is 4386.50 mg/l of COD, 190.4 mg/l of organic matter, and 330.50 mg/l for nitrate. These values exceed the Algerian standards (Table 3).

IV.2.2. Results of Effluent Treatment by Jujube Seeds

In order to examine the influence of the dose of jujube powder as a bio-coagulant on the efficiency of coagulation/flocculation, tests were conducted by varying the concentration from 0.625 to 12.5 g/L.

IV.2.2.1 The variation of pH and conductivity as a function of the bio-coagulant dose

pH is a very important factor to consider in the phenomenon of coagulation/ flocculation because it determines the state of the substances and the surface charge. The results obtained are shown in (Figure 6).

According to the obtained results, we notice that the pH values of the waters increase with the increase in the dose of coagulants. These obtained pH values remain within the Algerian standards for liquid effluents.

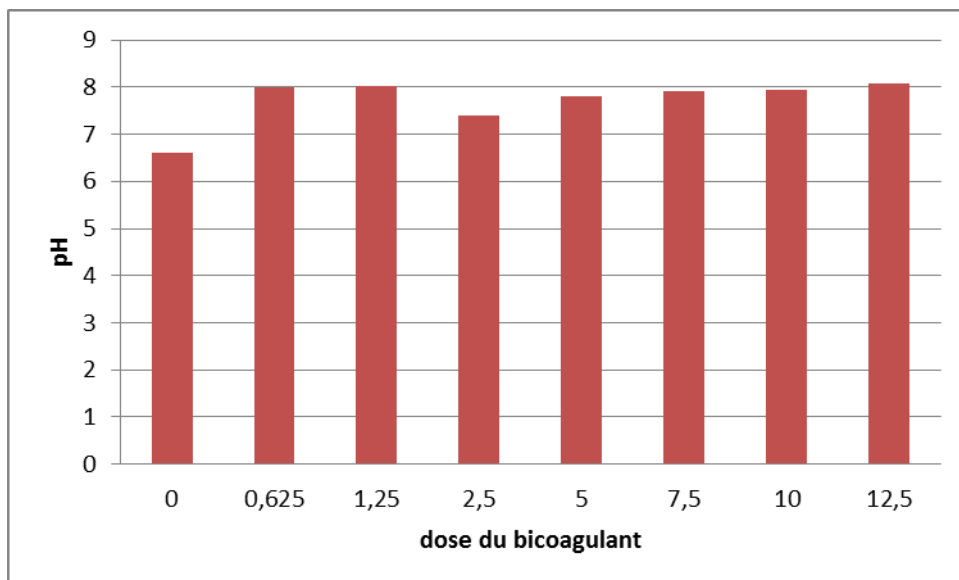


Figure 6: Effect of the bio-coagulant dose on pH variation

On the other hand, the values of electrical conductivity gradually decrease with the increase in the dose of all the coagulants used in the treatment, ranging from 25 mS/cm to 17 (Figure 7).

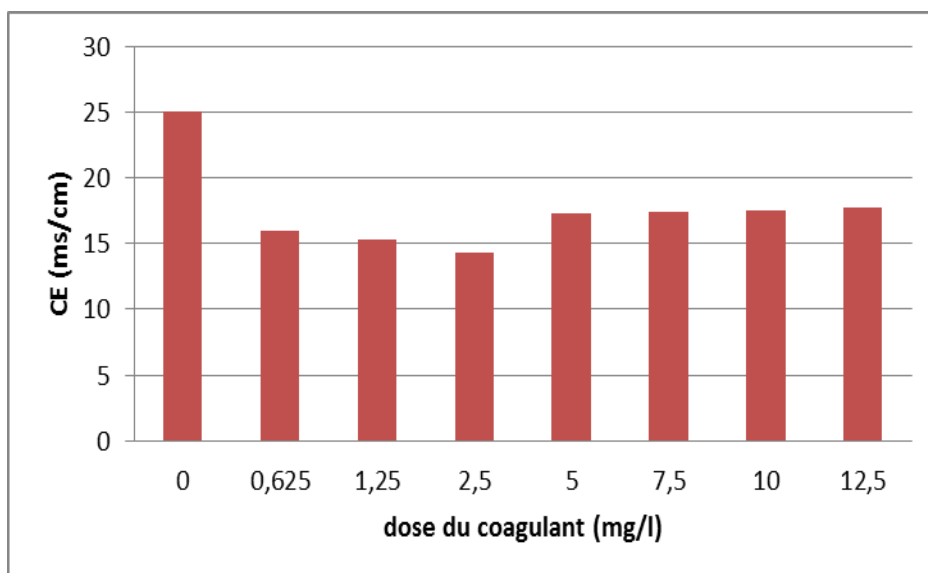


Figure 7: Effect of the bio-coagulant dose on the variation of conductivity

IV.2.2.2 Variations of chemical pollutants according to the dose of bio-coagulant

The monitoring of the variation in (COD, KMNO_4 Oxidizability) during the addition of increasing doses of the bio-coagulant in 1-liter beakers containing the effluent to be treated is presented in Figure 8. This figure shows two phases of variation, an initial phase of increasing pollutant removal rates followed by a phase of decreasing rates. The powder from jujube seeds has a significant effect at an effluent pH of 6.1. All the tests show that this biomaterial has coagulation/flocculation capacity, but with a low removal rate for oxidizable matter and phosphates compared to COD, TSS, and nitrates.

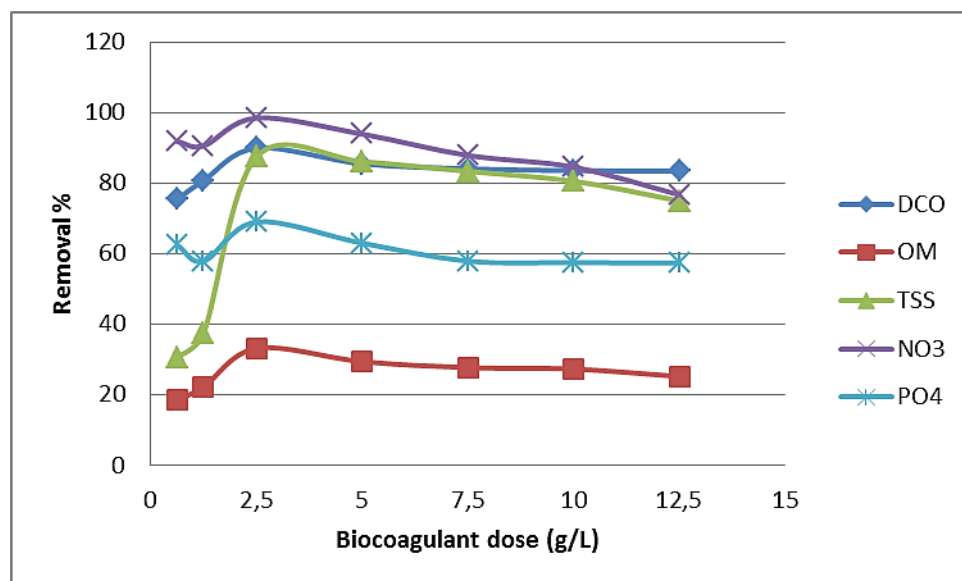


Figure 8: Effect of the bio-coagulant dose on the variation of COD, OM, TSS, PO₄, and NO₃

According to the results obtained, it was found that the removal of parameters (oxidizable matter, COD, TSS, phosphates, and nitrates) decreases with the increase in the coagulant dose. It was observed that the best removal efficiency is achieved with an optimal bio-coagulant concentration around 2.5g/l. It is observed that beyond this dose, the removal efficiencies of all parameters remain constant. This may be due to the saturation of the absorption sites of the studied pollutants. At this dosage, the removal efficiency of oxidizability, COD, TSS, phosphates, and nitrates varies respectively towards 33.2%, 90%, 87.5%, 70%, and 98.5%. These better removal rates are achieved at the acidic pH of the effluent. These results are very interesting since there is no need to add chemicals to adjust the pH of the effluent to be treated.

According to the previously mentioned results, we can provide some ideas regarding the operational mechanism for the removal of metals on jujube seeds.

The elimination of the studied pollutants can be explained by:

a- Either the large surface of jujube seeds comes into contact with the pollutants
b- Either to the functions of this surface that play an important role in cationic and anionic exchange operations as well as adsorption.

c- The surface of jujube pits, in the treated effluent which has an acidic pH (6.1) lower than its pH_{PZC} , is positively charged which promotes the adsorption of anionic species [27].

d- The chemical composition of jujube seeds (cationic proteins) plays the role of cross-linking the colloids in the effluent, as a means to neutralize their negative charge and weaken the rejection of the electrostatic double layer so that Van der Waals forces reduce the turbidity caused by suspended matter [30-32].

According to the study of the chemical composition of different parts of the jujube tree in Bechar, the results show that the jujube seeds in this region contain 20g of protein per 100g of seed mass [33].

The coagulation-flocculation process has revealed various mechanisms, often complex and highly dependent on the pH of the water to be treated, the coagulant dose, and the settling time.

This study is novel, there are no studies done on the removal of these wastewater pollutants by jujube seeds.

IV. CONCLUSION

According to the obtained results, the elimination of parameters (oxidizable matter, COD, TSS, phosphates and nitrates) decrease with the increase in the coagulant dose. The best removal efficiency is achieved with an optimal bio-coagulant concentration around 2.5g/l. It is observed that beyond this dose, the removal efficiencies of all parameters remain constant. This may be due to the saturation of the absorption sites of the studied pollutants. At this dosage, the removal efficiency of oxidizability OM, COD, TSS, phosphates, and nitrates varies respectively towards 33.20%, 90%, 87.5%, 70%, and 98.5%. These better removal efficiencies are achieved at the acidic pH of the effluent.

Laboratory tests as well as the results obtained in this study have made it possible to consider the hypothesis that it is possible to replace chemical coagulants with natural coagulants such as jujube seeds for the treatment of water loaded with organic and inorganic matter.

In perspective, in the liquid/solid separation by coagulation-flocculation, the choice of coagulants and/or flocculants is very broad and its influence is significant on the processes considered for carrying out the separation (sedimentation, flotation, and filtration) and for limiting the amount of sludge produced. Mineral and organic coagulants-flocculants are available. Knowledge of their chemistry, structure, and mechanisms of interaction with minerals is necessary to optimize the processes

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