

Potential of *Hibiscus Sabdariffa* and *Jatropha Curcas* as Natural Coagulants in the Treatment of Pharmaceutical Wastewater

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Abstract. Pharmaceutical wastewater is one of the most difficult wastewater to treat due to the presence of pharmaceutical compounds resulting in high concentration of organic matter, high turbidity and Chemical Oxygen Demand (COD). Chemical- based coagulation is a common method used to treat wastewater. However, the issue that has been raised with the use of chemical coagulants is their presence in water after treatment that can cause risks to the human health such as Alzheimer and cancer. Natural coagulants can be used as a safe alternative to these chemicals instead. Therefore, the objective of this experiment was to study the effect of *H. Sabdariffa* and *J. Curcas* as natural coagulants, separately and as a combination, on the treatment of pharmaceutical wastewater. Jar test experiment were carried out where beakers of 0.5L wastewater were mixed with the coagulants. The pH of the wastewater was varied from 2 to 12 while the coagulant dosage was varied from 40 to 200 mg/L. It was found that *H. Sabdariffa* works best at pH 4 and at a coagulant dosage of 190 mg/L with a highest turbidity removal of 35.8% and a decrease of COD by 29%. *J. Curcas* was found to perform best at pH 3 and with a coagulant dosage of 200 mg/L with a highest turbidity removal of 51% and a decrease of COD by 32%. When *J. Curcas* and *H. Sabdariffa* were used in combination, the optimum composition was found to be 80% *J. Curcas* and 20% *H. Sabdariffa* by weight with a maximum turbidity removal of 46.8% and a decrease in COD by 46%. In comparison between the two natural coagulants, *J. Curcas* is found to be a better and more suited coagulative agent for the treatment of pharmaceutical wastewater. The same experiment was carried with alum at pH 6 and coagulant dosage of 750 mg/L and a turbidity removal of 48% and a decrease in COD by 38% were recorded. In comparison with alum, *J. Curcas* was a better coagulant in treating the pharmaceutical wastewater. This shows that natural coagulants can be used to replace chemical coagulants in the treatment of pharmaceutical wastewater.

1 Introduction

In the last decade, traces of pharmaceuticals in the water cycle including drinking water have become a subject of discussion among the public, governments and the drinking-water regulators. Their presence in water, even at low concentrations, has raised concerns regarding its risks to human health from consuming these pharmaceuticals in drinking

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water [1], [2]. Therefore, it is essential to remove these pharmaceuticals from the wastewater before consumption. As a result, effective treatment methods must be applied to efficiently treat the contaminated water. Current pharmaceutical wastewater plants use methods such as activated sludge, membrane bioreactor and coagulation [3], [4].

Coagulation is a well-known technique used to reduce the pollutant contents in the water body that are present as turbidity, colour and organic matters [5], [6]. It is used as a treatment aiming to reduce the amount of pharmaceuticals such as paracetamol in the wastewater before it leaves as effluents [7]. Coagulants work by neutralising the charges caused by the colloidal particles. The suspended particles accumulate, forming flocs that then settle at the bottom and separate from the water suspension [6]. The most common coagulants used are chemicals such as the aluminium and ferric salts which are highly effective and cheap. However, recent studies have shown that traces of the aluminium based coagulants in the treated water can cause Alzheimer and other diseases such as intestinal constipation, abdominal colic and convulsions [6], [8]. Regarding the use of synthetic polymers, the presence of its residues in the water can be toxic and have strong carcinogenic properties [8]. Besides that, they are also ineffective in low-temperature water, produce large sludge volumes and significantly affect the pH of the treated water [9]. Therefore, researchers are focusing more on using natural coagulants which can be obtained from plants, animals or microorganisms as replacement to the chemical ones. They are non-toxic, biodegradable and can help to achieve the same results as the chemical coagulants [10].

Natural coagulants have been used for domestic households in the earlier days for traditional water treatment. However, due to the cheap cost and effectiveness of chemical coagulants, the use of natural coagulants was overlooked upon [10]. Furthermore, the use of natural coagulants have not been sufficiently investigated [10]. Therefore, this paper is focused on the study of *Hibiscus Sabdariffa* and *Jatropha Curcas* as natural coagulants. These plants are known as natural coagulants due to the presence of proteins in the seeds that act as an active coagulative agent [10]. These two plants are easily and readily accessible in various countries. The *H. Sabdariffa* seeds consist of 29.9% of proteins while *J. Curcas* seeds was found to contain 27.2% of proteins [11], [12]. Since the proteins inside the seeds act as a coagulative agent, both of these plants have the potential to treat wastewater.

H. Sabdariffa, most commonly known as Roselle, is used in herbal drinks, as food and as a flavouring agent in the food industry. The flower, known as calyx, is mostly used for these purposes. The seeds are most often discarded since till now, there is no use for it [11]. However, these seeds can be used to treat wastewater due to its coagulative properties. *J. Curcas* is a shrub or small tree belonging to the *Euphorbiaceae* family. Its main use is to extract oil from its fruit which is then used in the cosmetic industry, in soap making and as a substitute to kerosene or diesel. The seed and the press cake which is the waste after the oil extraction are normally discarded. However, these contain the protein which is the active coagulating agent that is efficient in the wastewater treatment [13], [14]. These wastes and the seeds can be used for coagulation, reducing the amount of waste and promoting sustainable development [8].

This paper investigates the efficiency of *H. Sabdariffa* and *J. Curcas* as natural coagulants, individually and as a combination, in the treatment of pharmaceutical

wastewater. The effect of pH and coagulant dosage on the coagulation process are also studied in this research. The performance of the natural coagulants is compared with the effectiveness of the chemical coagulant alum.

2 Materials and Methods

2.1 Materials

The materials used for this experiment include: good quality dry seeds of *H. Sabdariffa* and *Jatropha Curcas*, pharmaceuticals (namely paracetamol, ibuprofen, naproxen and caffeine) to simulate the wastewater, 1M hydrochloric acid and 1M sodium hydroxide (to adjust the pH of the wastewater), 0.5M sodium chloride solution (to extract the proteins from the seeds).

2.2 Preparation of synthetic wastewater

Synthetic pharmaceutical wastewater was prepared by dissolving pharmaceuticals in distilled water to simulate the pharmaceutical wastewater properties in terms of the concentration of pharmaceuticals present in the wastewater. The pharmaceuticals were crushed into a fine powder using a mortar and pestle. 62.5 mg of each pharmaceutical (paracetamol, ibuprofen, naproxen and caffeine) was added to 1L of distilled water to prepare the stock solution of the synthetic wastewater. The solution was mixed for 1 hour at 50 rpm to achieve uniform dilution of the pharmaceuticals. The synthetic wastewater was then allowed to settle for 24 hours for complete hydration before it was filtered and ready for the experiment.

pH of the wastewater was adjusted using 1M hydrochloric acid and 1M sodium hydroxide before the jar tests.

2.3 Preparation of the *H. Sabdariffa* and *J. Curcas* seed powder

Good quality seeds were selected and washed with tap water to remove any dust or impurities on the shells. They were then dried in an oven at $60 \pm 2^\circ\text{C}$ for 3 hours to remove all the moisture content from the seeds. The dried seeds were crushed into a fine powder using a mortar and pestle.

2.4 Preparation of the *H. Sabdariffa* and *J. Curcas* coagulant

To prepare the coagulants, sodium chloride was used as the solvent to extract the proteins from the seed powder. As studied by [13], sodium chloride was found to be the best solvent compared to distilled water and sodium hydroxide for the extraction of the coagulative agent from the seeds.

0.5g of the seed powder was dissolved in 100 ml of 0.5M sodium chloride. The mixture was blended for 2 minutes using a food processor. The solution was filtered through a muslin cloth and the filtrate was used in the following jar tests.

2.5 Jar Tests Experiment

The coagulation experiments were carried out by varying the pH, coagulant dosage and wastewater concentration in terms of the concentration of pharmaceuticals in the water. The pH was varied from 2 to 12 while the coagulant dosages were varied from 40 to 200 mg/L. The initial wastewater concentration was investigated at 62.5 mg/L of each pharmaceutical. The temperature of the wastewater was kept constant at room temperature.

The jar test was used to conduct the coagulation experiments. Six 0.5L beakers were each filled with 500 mL of wastewater. The optimum pH was first tested by varying the pH while keeping the coagulant dosage constant at 200mg/L. Upon addition of the coagulant, the mixture was stirred rapidly at 100 rpm for 4 minutes followed by slow mixing at 40 rpm for 25 minutes. The rapid mixing is to promote the collision between the pharmaceuticals and the coagulant while the slow mixing is to allow the growth of the flocs. The mixture was then allowed to settle for one hour for sedimentation, after which 50 ml of the liquid was carefully collected without disturbing the flocs settled at the bottom of the beakers. The liquid was then filtered and the residual turbidity (RT) and COD of the filtrate were measured. From the results, the pH with the best turbidity and COD removal was chosen as the optimum pH.

The same procedures were repeated while experimenting with different coagulant dosages while keeping the pH constant at the optimum value. The COD and turbidity are measured and the dosage with the best turbidity and COD removal was taken as the optimum. Next, the initial wastewater concentration was varied while the pH and the dosage was kept to their optimum values. The initial wastewater concentration with the best turbidity and COD removal was selected. The same coagulation test without the addition of coagulants was carried out as a control and the residual turbidity (RTc) and the COD (CODc) were measured.

The jar coagulation tests were carried out for both J. Curcas and H. Sabdariffa individually and as a combination.

2.6 Analytical Method

When the pharmaceutical wastewater is put under study, different parameters are always considered to have a better indication of the characteristics of the wastewater. The parameters measured are pH, COD, Biological Oxygen Demand (BOD), turbidity, total suspended solids (TSS) and total dissolved solids (TDS) [15].

As mentioned earlier in Section 1, coagulation is used to reduce the amount of colloidal matter in the water. Turbidity measures the cloudiness of the water, and therefore measures the amount of colloidal matter present inside the water.

Coagulation is a treatment used prior to the biological treatment which involves activated sludge where microorganisms are used to treat the water [16]. During coagulation, a reduction of COD is favored so that when the treated water undergoes the biological treatment, less sludge is produced from the microbial activity. As a result, the two parameters measured in this study are turbidity and COD.

Turbidity

It was measured using a turbidimeter (EUTECH Instruments TN-100). The turbidity removal was calculated as shown by Eq.1:

$$\text{Turbidity removal} = \frac{RT_c - RT}{RT_c} \times 100 \% \quad (1)$$

Where: (RT_c) is the turbidity of the wastewater before undergoing coagulation, (RT) is the turbidity of the wastewater after undergoing coagulation.

Chemical Oxygen Demand (COD)

COD was measured using reaction cells of range 25-1500 mg/L which contained sulphuric acid, potassium dichromate and mercury (II) sulphate as reagents. 3 mL of the sample to be tested was carefully added to the reaction cells inside a fume hood. The cell was then vigorously shaken and heated at 150 °C for 2 hours using a thermoreactor. The cells were then removed and let to cool to room temperature. The COD was then measured using a photometer (Prove 300 UV/VIS Spectrophotometer). The COD removal was calculated as shown by Eq.2:

$$\text{COD removal} = \frac{COD_c - COD_i}{COD_c} \times 100 \% \quad (2)$$

Where: (COD_c) is the COD of the wastewater before undergoing coagulation, (COD_i) is the COD of the wastewater after undergoing coagulation.

3 Results & Discussion

The synthetic wastewater was prepared to simulate the properties of the real pharmaceutical wastewater. The properties of the synthetic wastewater prepared are shown in Table 1 below:

Table 1: Characteristics of synthetic wastewater.

Characteristics of the synthetic wastewater	
Temperature (°C)	30
pH	4.95
Turbidity (NTU)	9.96
COD (mg/L)	665

The pharmaceutical compounds used to simulate the wastewater are acidic in nature and as a result, the wastewater becomes slightly acidic with an average pH of 4.95. The real wastewater is also acidic due to the acidic nature of the pharmaceutical compounds present [15].

3.1 Coagulation activity using *H. Sabdariffa*

3.1.1 Effect of pH on coagulation activity

Fig. 1 and Fig. 2 shows the effect of pH on the turbidity and COD of the treated water after jar tests. pH is an important factor to consider since the solubility of the matter depends on the pH level of the wastewater. The wastewater consists of mainly negative charges and the pH at which the particles become stable is known as the isoelectric point [6]. The pH is varied from 2 to 12 while the coagulant dosage is kept constant at 200 mg/L. From the results obtained, it can be seen that the lowest turbidity is obtained at pH 4 with a turbidity removal of 44.7% and a decrease in COD by 22.9%. This is in agreement with a previous study done by [6] in which it was found that *H. Sabdariffa* is efficient under acidic conditions, especially at pH 4.

This phenomena can be explained by studying the amino acids present in the *H. Sabdariffa* seeds and their isoelectric point. The coagulative agent present in the seeds consists mainly of glutamic acid and aspartic acid which are categorised as acidic amino acids [17]. While the isoelectric point of glutamic acid and aspartic acid are 3.2 and 2.77 respectively, the seeds also contain significant amount of the other amino acids with isoelectric point ranging from 3 to 6 as shown earlier in table 3. As a result, the coagulative agent works best in acidic conditions, especially at pH 3 and 4.

Also, from Fig. 1, pH 4 gives the lowest turbidity of 5.51 NTU with a turbidity removal of 44.7%. It can also be seen that at some pH values, the turbidity increases above its initial turbidity of 9.96 NTU. This is because the pharmaceutical wastewater can only be treated effectively at its optimum pH and at other pH far from its optimum pH, the coagulation activity fails and is unable to produce any flocs. As a result, it causes an increase in the turbidity of the water.

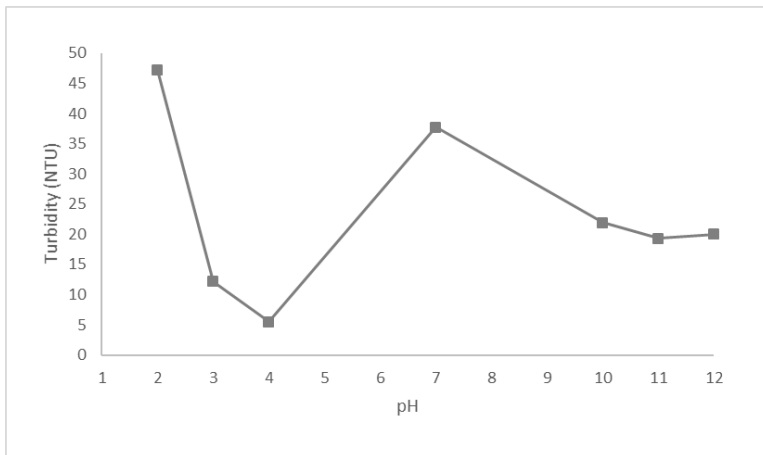


Fig. 1. The effect of pH on the turbidity of the wastewater after coagulation.

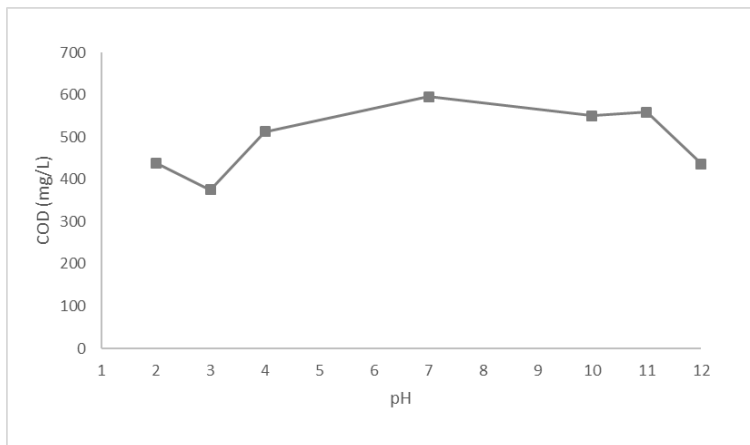


Fig. 2. The trend in COD when varying the pH of the wastewater after coagulation.

Fig. 1 and 2 show the relationship between turbidity and COD. The graph for COD shows a similar trend to the graph of turbidity. This shows that pH also has an effect on the COD of the wastewater. At pH 4, the % COD removal is 22.9%.

3.1.2 Effect of coagulant dosage on coagulation activity

The formation of flocs depends on the coagulant dosage. The charges of the coagulants work by neutralising the charged ions in the synthetic wastewater. The floc strength depends on the attraction and repulsion between the particles. When the dosage is low, the particles do not have enough energy and therefore form weak and small flocs [18]. Overdosing also result in poor performance of coagulation and also directly increases the cost of dosing and sludge formation [19]. Therefore, the coagulant dosage was varied from 40 to 200 mg/L.

From Fig.3, it is found that at 190 mg/L, the coagulant gives the highest turbidity removal of 28.4% with a COD removal of 28.3%. It can be seen that at lower dosages, the turbidity removal is low due to insufficient dosages producing weak flocs that are unable to settle down. This is also in correlation with the findings of [20]. As from 90mg/L of coagulant, the COD remains stable at 476 mg/L. This shows that the dosage does not have much of an effect on the COD of the water.

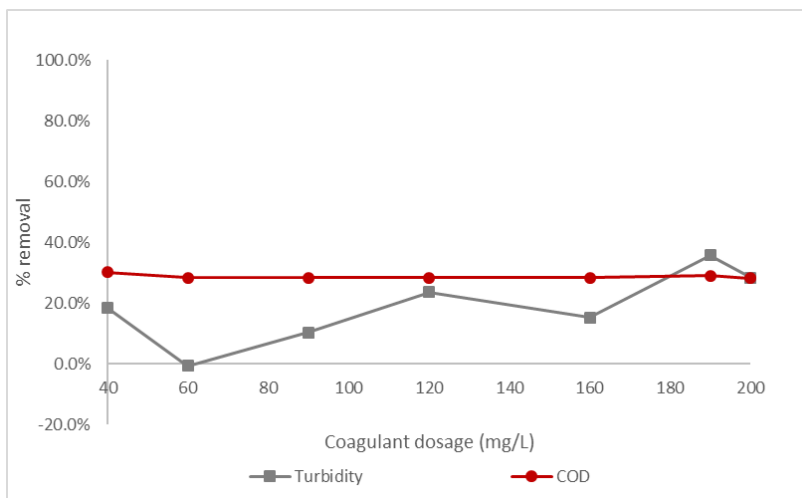


Fig. 3. Percentage removal of turbidity and COD with increasing coagulant dosage.

3.2 Coagulation activity using *J. Curcas*

3.2.1 Effect of pH on coagulation activity

Fig.4 and Fig.5 show a similar trend with the effect of increasing pH. The lowest turbidity is observed at pH 3 with a turbidity removal of 41% and COD removal of 34.9%. The turbidity is reduced from 9.96 NTU to 5.88 NTU. This is in agreement with [13] where it proves that *Jatropha* works best in acidic conditions and works best at pH 3.

It can also be observed that only at pH 3 and 4, the coagulative agent is able to reduce the turbidity. This is because at pH 3 and 4, the positive charges on the amino acids that make up the protein molecules dominate. The composition of amino acids in the seeds of *J. Curcas* was studied by [21]. Glutamic acid and aspartic acid were abundantly found in the seeds. Since these amino acids are acidic and have an isoelectric point at 3.22 and 2.77 respectively, they favour coagulation in acidic conditions, especially at pH 3 [22]. The *J. Curcas* protein is expected to work well as a cationic coagulant agent. As an amphoteric molecule, the charge of the amino acids is dependent on the pH of the environment. As a result, this is why we can observe flocs at pH 3 and 4 [23]. However, at pH greater than 3, the positive and negative charges of the different amino acids in the protein may have

reduced the cationic potential of the coagulant and as a result, no coagulation can be observed at these pH [23].

However at other pH values, *J. Curcas* is unable to neutralise the charges in the wastewater and unable to produce any flocs. This may be due to the presence of oil in the jatropha seed powder which was not extracted prior to coagulation. The oil when mixed with the sodium chloride solvent produces a milky emulsion that causes an increase in turbidity at pH 2 and at alkaline conditions [14].

Similar to the coagulation activity with *H. Sabdariffa*, the COD of the treated water shows a similar trend as the turbidity of the water. When the turbidity decreases, the COD of the water also reduces. Therefore, this shows that pH also has an effect on the COD of the water.

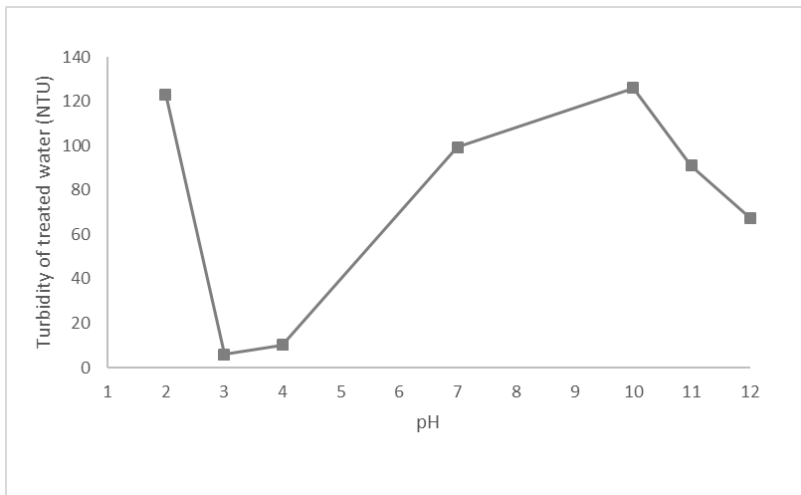


Fig. 4. The effect of pH on the turbidity of the wastewater after coagulation.

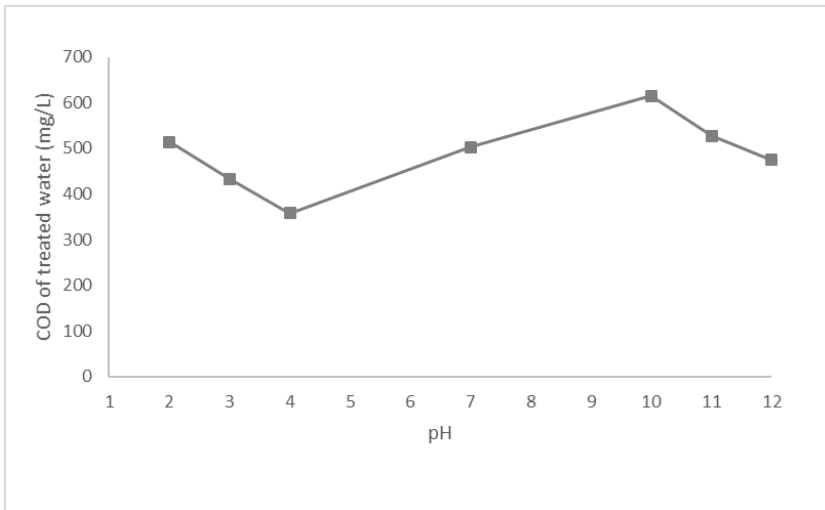


Fig. 5. The effect of pH on the COD of the wastewater after coagulation.

3.2.2 Effect of coagulant dosage on coagulation activity

From Fig. 6, it can be seen that the turbidity increases when the coagulant dosage is below 80 mg/L. This is because the dosage is not enough to produce any flocs and therefore the milky emulsion of the jatropha coagulative agent causes the turbidity of the water to increase. From 90mg/L onwards, the turbidity considerably decreases and reaches its lowest turbidity of 4.81 NTU at 200mg/L. The highest turbidity reaches a 51% removal with a 32% decrease in COD.

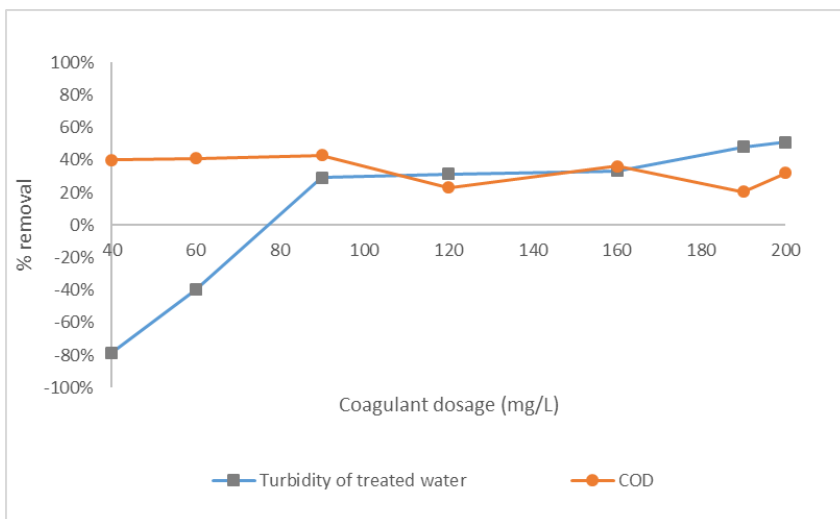


Fig. 6. The effect of coagulant dosage on the turbidity and COD % removal.

3.3 Coagulation activity using a combination of *H. Sabdariffa* and *J. Curcas*

3.3.1 Effect of pH on coagulation activity

The effect of pH is studied on treatment of water by using a combination of both seeds. As mentioned in Section 3.1.1, *H. Sabdariffa* works best at pH 4 while in Section 3.2.1, it was found that *J. Curcas* works at an optimum pH of 3. For a combination of both, it is expected that the coagulants will work under acidic conditions as well. Therefore, the pH is varied from 2 to 7 while the combination is kept to 200 mg/L with equal amounts of *J. Curcas* and *H. Sabdariffa* (50:50%).

Fig. 7 studies the effect of pH on the turbidity of wastewater after coagulation. It can be observed that the turbidity considerably decreases at pH 3 and 4. It reaches its lowest turbidity of 7.59 NTU at pH 3 with a turbidity removal of 24% and a 32.3 % decrease in COD. Fig. 8 shows that pH also affects the COD of the wastewater. The highest decrease in COD is observed at pH 3 and pH 4.

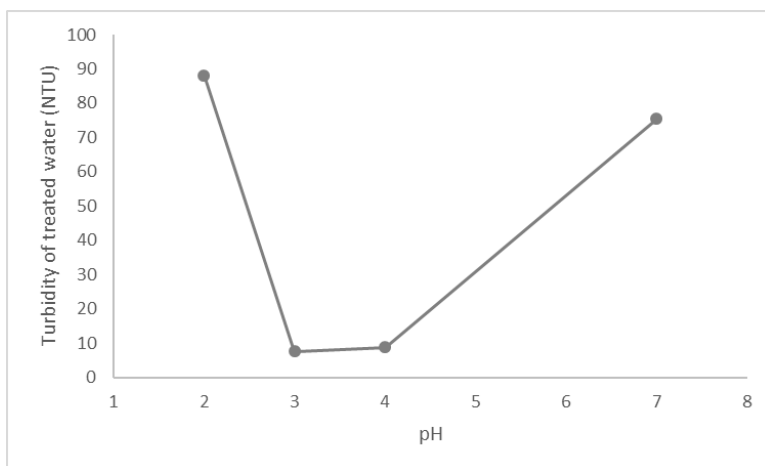


Fig. 7. The effect of pH on the turbidity of treated water after coagulation.

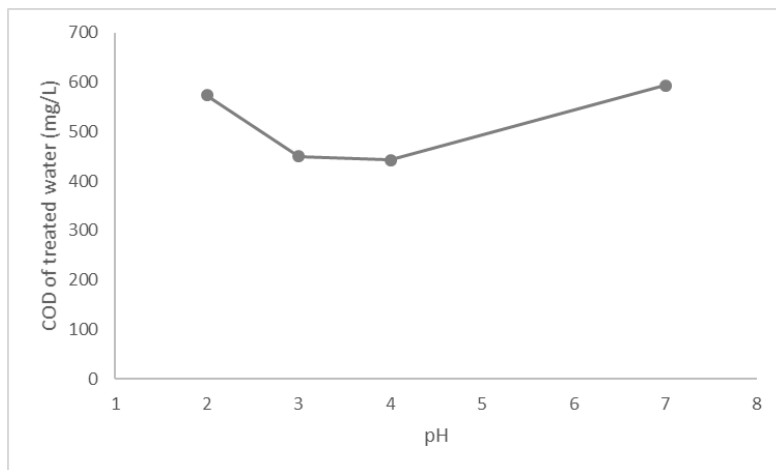


Fig. 8. The effect of pH on the COD of treated water after coagulation.

3.3.2 Effect of coagulant dosage on coagulation activity

Fig. 9 shows the effect of increasing the weight of *J. Curcas* in the mixture of both coagulants on the turbidity and COD of the treated water after coagulation. It can be observed that the % turbidity removal gradually increases from 38.8% to 46.8% when the % weight of *J. Curcas* increases. However, the COD remains almost constant at a COD removal of 46%. This shows that increasing the amount of *J. Curcas* does not affect the COD of the water. The optimum composition is 80% *J. Curcas* and 20% *H. Sabdariffa* by weight with a maximum turbidity removal of 46.8% and a decrease in COD by 46%. This shows that *J. Curcas* has a higher effect than *H. Sabdariffa* in the treatment of the wastewater.

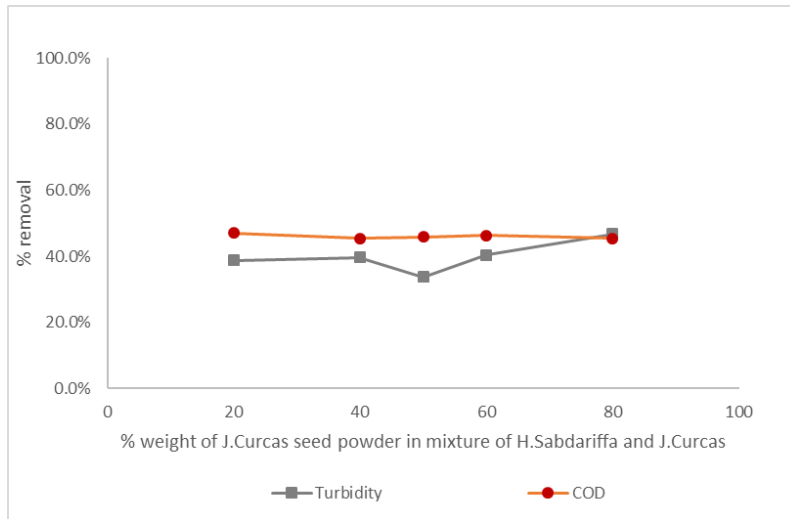


Fig. 9. The effect of coagulant dosage on the turbidity and COD of the wastewater.

3.4 Coagulation with Alum

The results are compared with the conventional chemical coagulant which is alum. The effect of alum in the treatment of pharmaceutical wastewater was studied by [7]. It was found that alum worked best at pH 6 with a dosage of 750mg/L.

The synthetic wastewater was treated with alum with the optimum conditioned mentioned above. The same experiment was carried out, with only alum being used as the coagulant. The chemical coagulant was successful in treating the pharmaceutical wastewater with a turbidity removal of 48% and a COD removal of 53%.

3.5 Comparison of results for all different coagulants experimented

The results obtained for all different scenarios tested in this research are compared. The graph below shows the results obtained for all 4 scenarios.

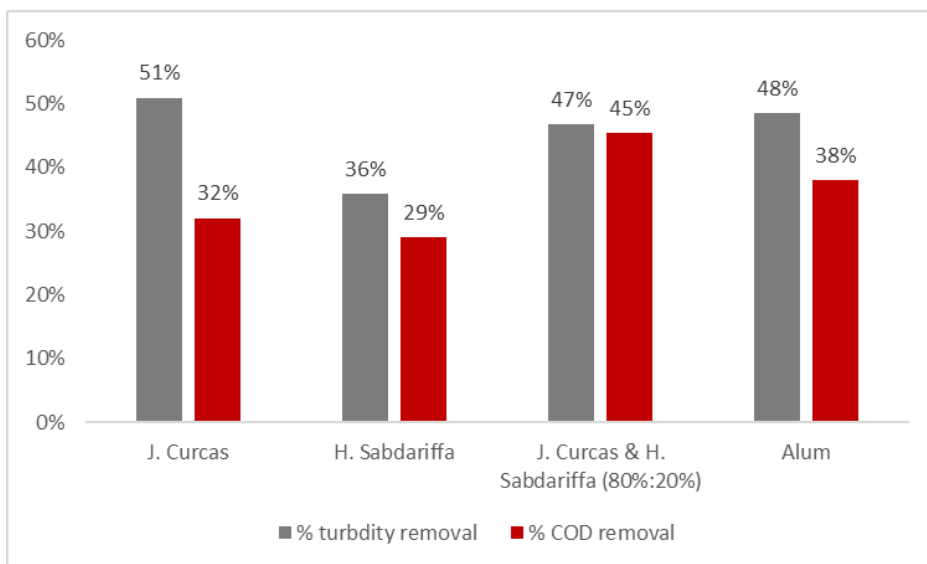


Fig. 10. Result comparison of all the different scenarios tested.

From Fig. 10, it can be seen that in terms of % turbidity removal, *J. Curcas* gives the best results compared to the others. It is followed by Alum with a 48% turbidity removal. *H. Sabdariffa* gives the lowest results among the 3 coagulants. This shows that the natural coagulants can be used to replace chemical coagulants in the treatment of pharmaceutical wastewater since the results obtained are comparable and close to each other. By comparing both natural coagulants, *J. Curcas* is the most suitable one to be used as it is more effective in treating the wastewater.

4 Conclusion

In this study, it was found that both *H. Sabdariffa* and *J. Curcas* were able to effectively treat pharmaceutical wastewater. It was found that *H. Sabdariffa* works best at pH 4 and at a coagulant dosage of 190 mg/L with a highest turbidity removal of 35.8% and a decrease of COD by 29%. *J. Curcas* was found to perform best at pH 3 and with a coagulant dosage of 200 mg/L with a highest turbidity removal of 51% and a decrease of COD by 32%. In comparison, *J. Curcas* is found to be a better and more suited coagulative agent for the treatment of pharmaceutical wastewater. Even a combination of both coagulants only gave a turbidity removal of 46.8%. This study also showed that only pH has a distinctive effect on the COD of the wastewater while both pH and dosage has an effect on the turbidity of the wastewater. In comparison with alum, *J. Curcas* gave a better result in the treatment of

the wastewater. This research was successful in showing that natural coagulants can be used to treatment of pharmaceutical wastewater and therefore can replace the use in alum in treatment plants.

For future work, the oil can be extracted from the *J. Curcas* seeds prior to coagulation. Also, coagulation of higher concentrations of pharmaceutical wastewater can be studied. High Performance Liquid Chromatography (HPLC) can be used to detect the pharmaceuticals still present in the wastewater after coagulation and its corresponding concentration.

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