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A NOVEL BIO-COAGULANT AID TO TREAT HIGH TURBID WATER CONTAINING HUMIC ACIDS

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ABSTRACT

In general, the production of potable water from raw water sources involves the coagulation or flocculation process to remove turbidity. Alum has been widely used as a coagulant; however, there is a concern about its associated risk of Alzheimer's disease. As a result, there has been considerable interest in the development of natural coagulants and coagulant aids in order to reduce the dose of alum. This study aimed at evaluating the use of Corchorus Olitorius L. (COL), a leafy vegetable grown in Africa and the Middle East, as a novel coagulant aid over other synthetic coagulant aids. It is a widely produced agricultural waste and does not require further chemical treatment. Various tests were carried out to evaluate the optimal dosage and conditions required to achieve the optimum removal of both turbidity and humic acid from water. Based on the results of the jar test, COL was found to be an efficient coagulation aid. It has the ability to reduce the primary coagulant dose (from 600 mg/L to 300 mg/L) and residual turbidity (from 5.63 to 2.3 NTU). This novel coagulant aid reduced the concentration of total organic carbon to the zero level and increased the rate of flocculation.

Keywords: *Alum, Corchorus Olitorius L., Coagulation, Flocculation, Turbidity, Water Treatment.*

1. INTRODUCTION

The water from surface or ground sources is not clean enough for consumption as it contains particles of the colloidal suspension. These particles cannot be separated using conventional physical methods, such as filtration or settling, unless they are agglomerated through processes like coagulation and flocculation. The colloids commonly found in the wastewater are stable because of their electrical charge (positive or negative). However, most colloidal particles in the wastewater possess a negative charge (Farajnezhad and Gharbani, 2012). The coagulation process is effective in removing high concentrations of organic pollutants, heavy metals, and certain anions. It is also effective in reducing the color of particles, chemical oxygen demand (COD), and total suspended solids (TSS). Aluminum salts are the most widely used coagulants; however, they may cause numerous neurological disorders in humans (Tassinari et al., 2013). It has been reported that iron salts can be more efficient and cheaper than the aluminum salts, but their high importation cost is seen as a serious setback for the developing countries

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(Ugonabo et al., 2012). One of the major disadvantages related to the use of coagulants is the huge amount of toxic sludge produced during the process (Zemmouri et al., 2012).

There are two main types of coagulants, namely primary coagulants and coagulant aids, used in the water treatment process. The role of primary coagulants is to neutralize the electrical charges of the colloidal particles in the water, which causes the particles to clump together. Comparatively, the addition of coagulant aids increases the floc's density, adds roughness to the flocs so that they do not break up during the mixing and settling process, and reduces the flocculation and settling time (Aziz et al., 2007).

Lime and bentonite, clay minerals, activated Si O_2, and cationic or anionic polyelectrolytes are some examples of coagulant aids (Cheng et al., 2008). Previous studies have investigated the use of tannin as a primary coagulant and coagulant aid in water treatment (Özacar and Şengil, 2002). Chitosan has also been investigated as a coagulant or flocculent for a wide variety of suspensions (Zemmouri et al., 2012; Hu et al., 2013). A new coagulant aid, Enteromorpha extract, was also investigated for enhancement of the coagulation performance (Zhao et al., 2013).

The most important benefit of using a coagulant aid is the reduction in the amount of a coagulant used in any given case, which in turn reduces the sludge and chemical residuals present in the drinking water. In an attempt to avoid the hazards of using metal salts as coagulants, more interest has been generated towards the development of natural coagulants and coagulant aids (Amud and Amoo, 2007; Ugonabo et al., 2012; Hu et al., 2013; Yang et al., 2013, Choy et al., 2014). However, most of the natural coagulants and coagulant aids are very expensive. Hence, it has become necessary to develop non-toxic, efficient, and low-cost coagulant aids (Zhao et al., 2013). The natural coagulants may consist of plant seeds, leaves, roots, and animal extracts (Gunaratna et al., 2007, Fedala et al., 2015). Other natural substances tried as coagulant aids include Moringa olifera (drumstick tree), red bean, and red maize (Gunaratna et al., 2007; Poumaye et al., 2012). The performance of agro-based materials, such as Surjana seeds, Nirmali seeds, and maize, as coagulant aids with alum as a coagulant was investigated by Raghuwanshi et al. (2002).

For any new coagulant/coagulant aid to be considered, it must be efficient, cost-effective, sustainable, and environmentally safe.

Corchorus olitorius L. (COL) is a leafy vegetable grown in Africa and the Middle East. It is native to Egypt and popular in the Mediterranean regions, Japan and Southeastern Asia. The young green leaves and shoots of this vegetable can be picked and cooked. The stems and roots are considered as agricultural wastes. COL possesses a wide spectrum of medicinal applications. Its leaves and roots are used as herbal medicines. Traditionally, its leaves have been used for the treatment of pain, fever, chronic cystitis, and tumors. Its seeds have been reported to possess estrogenic activity (Oyedeji and Bolarinwa, 2013), and it also induces wound healing and regenerates injured tissues (Barku et al., 2013). Apart from these advantages, a thorough literature survey indicates that COL has not been used as a coagulant or coagulant aid.

The aim of this study is to investigate the use of COL waste as a coagulant aid to enhance the turbidity and total organic carbon (TOC) removal and accelerate the flocculation time. Operating variables that were investigated included turbidity, pH, coagulant and

coagulant aid dosages, slow mixing time, and sedimentation time. A comparison was also drawn between the treatment methods, with or without filtration. The COL coagulant aid was optimized on the basis of TOC removal, residual turbidity, and flocculation time.

2. MATERIALS AND METHODS

2.1. MATERIALS

The kaolin stock solution (turbid water) was prepared by dissolving 10 g of kaolin (Acros Organics, Gee, Belgium) in 1 liter of distilled water. The solution was stirred for 24 hours for complete hydration of kaolin particles. The turbid water was prepared by adding part of the stock solution to distilled water to reach the required turbidity. The alum solution (Panreac Quimica SA, Spain) was prepared by dissolving 10 g of the solid salt in 1 liter of distilled water. This solution was used as a coagulant without any further dilution. The coagulant aid was freshly obtained from the local market of Saudi Arabia. The stems were cut into small pieces and crushed with the help of a mortar and pestle. The liquid extract was collected and kept in a glass container. The working solution was prepared by dissolving 1 g of this extract in 1 liter of distilled water. The humic acid solution was prepared by dissolving 1 g of the solid material (LobaChemiw, India) in 1 liter of distilled water. A suitable volume of humic acid solution was added to reach the required humic acid concentration in the turbid water (as illustrated in Figure 6).

2.2. COAGULATION EXPERIMENTS

The coagulation or flocculation tests were conducted using a conventional jar test apparatus equipped with six beakers of 1000 mL volume. The turbid water samples (100 NTU) were stirred for 1 min to assure the homogeneity of the turbid water. The beakers were filled with 500 mL of turbid water for each test run. No adjustment of pH was performed unless when evaluating the effect of pH on the coagulation process. Calculated amounts of coagulant were added to each beaker to reach the required dose. Care was taken not to change the total volume of the turbid water. Rapid mixing was performed at 250 rpm for 1 min followed by slow mixing at 50 rpm for 20 min. The flocs formed were then allowed to settle for 40 min. After the settling period, the samples were withdrawn from the supernatant for further analysis. All the experiments were carried out at an ambient temperature of 20–22 °C. The different factors affecting the coagulation process were studied by changing one factor at a time while keeping the other factors constant. For instance, to study the effect of initial pH, all other factors were kept constant, and the pH of the turbid water was adjusted to different values by using either 1 N H_2SO_4 or 1 N NaOH. The experimental conditions are summarized in Table 1.

TABLE 1: EXPERIMENTAL CONDITIONS OF THE COAGULATION/FLOCCULATION EXPERIMENTS

	Rapid mixing time,min / rate,rpm	Slow mixing time,min / rate,rpm	pH of turbid water	Dose of alum as mg/L $Al_2O_3.18H_2O$	Dose of COL, mg/L
Effect of alum as the primary coagulant dose	1/250	20/50	7.5	20-1800	-
Effect of COL as the primary coagulant dose	1/250	20/50	7.5	-	2-200
Effect of pH when using alum	1/250	20/50	1-11	600	-
Effect of pH when using alum+COL	1/250	20/50	7.5	300	3
Effect of COL as coagulant aid on removal of turbidity	1/250	20/50	7.5	20-600	2-200
Optimization of alum dose and coagulant doses for removal of humic acids	1/250	20/50	7.5	20-600	2-10
Effect of coagulant aid on the flocculation time	1/250	20/50	7.5	300	3

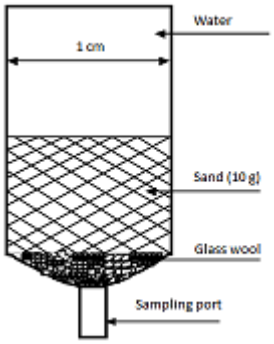

Fig.1. Schematic diagram of the filtration column

The filtration was performed in a column of 60 cm height and 1 cm diameter. Then 10 g of sand was placed in the column and was supported by glass wool. The sand was introduced to the bed without stratification. After the introduction of sand, gentle tapping was applied on the outer surface of the column to avoid possible channeling through the sand bed. A schematic diagram of the filtration column is depicted in Figure 1. The supernatant from the coagulation or flocculation process was introduced into the column by using a peristaltic pump. The effluent from the column was collected, and the residual turbidity was measured. Duplicates of the experiments were conducted, and the average was taken.

2.3. Analytical Method

The TOC representing the humic acid and residual coagulant aid was determined by following the Hach Method 10129 of the Hach Water Analysis Handbook (Hack 1997), using DRB200 Reactor and DPR 890 colorimeter. Turbidity was measured using the calibrated Nephelometer (Hack Turbidity Meter Model 2100), and pH was measured using pH 501, EuTech Instruments.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Sand bed, turbid water, and COL

The characteristics of the sand bed used were d10 (the grain diameter at 10% passing), d60 (the grain diameter at 60% passing), and uniformity coefficients (UC) (d60/d10) of the sand bed were 0.173 mm, 0.433 mm, respectively, giving a ratio of 2.5. The turbid water characteristics are illustrated in Table 2a while the characteristics of COL are introduced in Table 2b.

Table 2a: Characteristics of turbid water

TOC, mg/L	pH	Turbidity, NTU	EC, S/m	TDS, mg/L
50	7.5	100	0.204	102

EC: Electrical conductivity; TOC: Total carbon content; TDS: Total dissolved solids

Table 2b: Characteristics of Corchorus olitorius L. (Ndovu and Afolayan, 2008)

	Total carbohydrate, mg/g	Crude protein, mg/g	Crude fiber, mg/g	Crude lipid, mg/g	Ash, mg/g
Leaves	695.0±32.4	162±3.4	20.3±1.0	17.2±2.9	105.2±1.0
Stems	802.0±18.2	51.0±1.8	88.2±4.8	69.0±1.3	51.9±1.2

3.2. Coagulation performances of separate coagulant and coagulant aid

The effect of alum dosages on the turbidity removal efficiency is shown in Figure 2A. It is apparent from the results that with an increase in the alum dose from 20 to 300 mg/L, the turbidity removal efficiency decreased from 93.5% to 88.3%, respectively. Further increase in the alum dose to 600 mg/L caused an increase in the turbidity removal to 94.4%. After this dose, the turbidity removal showed a declining trend. Thus, the highest turbidity removal efficiency (94.4%) equivalent to the final turbidity of 5.6 NTU was obtained at an alum dose of 600 mg/L, which was considered as the optimum dose of alum under these conditions. The initial increase in turbidity of the water samples after adding the coagulant may be attributed to the coagulant itself, which added turbidity to the water before starting its coagulating action. At high coagulant doses, aluminum hydroxide is produced, and suspended solids causing turbidity are removed by entrapment into or sorption onto the hydroxide flocs (Demirci et al., 1998).

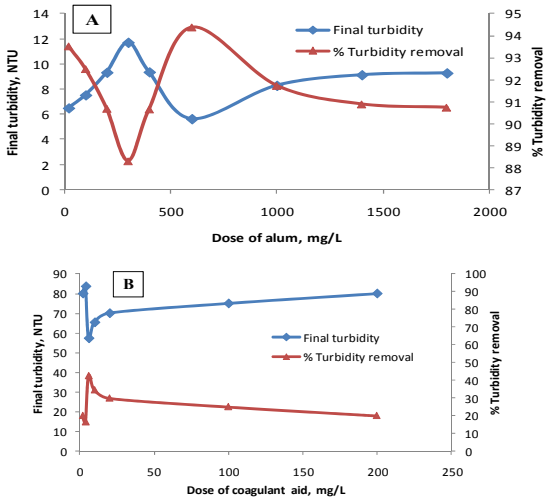

Fig.2. Effect of coagulant or coagulant aid doses on removal of turbidity; A: alum, B: COL

COL was tried as the primary coagulant in a separate experiment. As illustrated in Figure 2B, the different doses of COL resulted in poor turbidity removal efficiency compared to the alum. The highest efficiency that could be attained was 42.5%. The lowest final turbidity was 57.5 NTU at a dose of 6 mg/L of COL. An explanation is a steric hindrance that may take place between the polymer molecules at high concentration (Hu et al., 2013). The poor performance of natural polymers when used as primary coagulants has been reported by other researchers (Hu et al., 2013; Zhao et al., 2013).

The data presented in Table 2b indicates the presence of high concentrations of both carbohydrates and lipids in the stems. The presence of these constituents increases the content of organic matter in the treated water that represents a disadvantage due to the increase in turbidity in certain situations if the dose of the primary coagulant is not well adjusted.

3.3. Effect of turbid water pH on turbidity removal

In the coagulation–flocculation process, pH is a very important factor since the coagulation occurs within a specific pH range for a particular coagulant. When alum is added to water, a series of soluble hydrolysis species are formed. The species that may be produced includes Al(OH)3, Al3+, Al(OH)2+, Al(OH)2+, and Al(OH)4- ions. These hydrolytic species have positive or negative charges depending on the pH of the water. The positively charged hydrolytic species can adsorb onto the surface of colloidal particles and destabilize them. The equilibrium state between the concentrations of the above ions depends on the pH of the solution (Libecki and Dziejowski, 2008). The following equilibrium is obtained shifting to the right with increasing pH:

$$Al^{3+} \rightarrow Al(OH)^{2+} \rightarrow Al(OH)_2^{+} \rightarrow Al(OH)_3 \rightarrow Al(OH)_4^{-}$$

The coagulation process with alum as the sole coagulant is capable of achieving significant turbidity removal that is highly pH-dependent (Yan et al., 2008; Yang et al., 2010). As shown in Figure 3A, the removal efficiency with alum decreased with the increase in the pH from 1 to 3 and then increased with increasing pH reaching a considerably high level of 7.1. Further increase in the pH led to slightly decreased removal efficiency. At pH 11, the coagulation efficiency almost reached the slightly high removal value of 97.1% (final turbidity of 2.9 NTU). At this high pH, high precipitation of aluminum will take place leading to the removal of turbidity by the sweep-floc mechanism (Hayrapetyan et al., 2006; Libecki and Dziejowski, 2008). In our tested samples, the higher efficiency of turbidity removal at pH 1 may be attributed to the removal of humic acids, followed by kaolin turbidity. The high turbidity removal at higher pH may be attributed to the association of humic acids with other suspended solids during the flocculation.

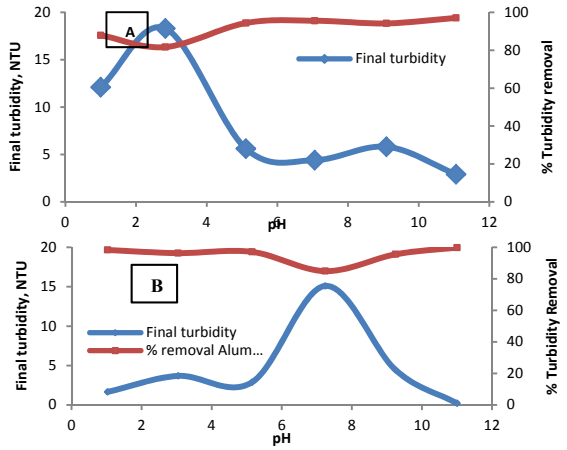

Fig.3. Effect of water pH on removal of turbidity; A: alum, B: Alum + aid. Alum dose is 300 mg/L and COL DOS IS 3 mg/L

On the other hand, when COL was added as a coagulant aid, as illustrated in Figure 3B, a slightly different behavior was observed. Increasing the pH led to a clear decrease in the removal efficiency. The removal efficiency decreased from a value of 98% at pH 1 to 85% at pH 7.5. It was clear that the removal efficiency of using COL as a coagulant aid has been enhanced by the effect of pH. A final turbidity of 0.214 NTU was attained at pH 11. This value is very low as compared to that obtained when using alum alone, and it is better than the value recommended by World Health Organization (WHO) (1 NTU).

3.4. Effect of coagulant aid on turbidity removal

Two separate experiments were performed. In the first experiment, one set of beakers containing turbid water was treated with alum alone. Another set was treated by adding the same dose of alum and different doses of COL. In the second experiment, the dose of alum was kept constant at its optimum value, with varied doses of COL from one beaker to another. For the second set, the dose of alum was varied, and the dose of COL was kept constant at its optimum value. As illustrated in Figure 4, the addition of the coagulant aid was very influential in decreasing the turbidity. The results indicated that a small dosage of the coagulant aid (3 mg/L) decreased the water turbidity from 5.63 to 2.3 NTU. The later value of turbidity would not have been achieved without using the coagulant aid, even under optimum conditions.

Figure 5 represents the optimization conditions for both the alum and COL. It is clear that the optimum doses of both alum and COL were 300 mg/L and 3 mg/L, respectively. The correct selection of the optimum combination doses of coagulant and coagulant aid resulted in 50% decrease in the initial primary coagulant dose. Moreover, the final turbidity has been enhanced to reach a value of 2.3 NTU compared to a value of 5.63 NTU when alum was used alone. As illustrated in Table 2b, the stem of the plant contains a substantial amount of proteins. These proteins may impart the positive charge to the solution that will lead to their binding with negatively charged particulates, causing turbidity (Tassinari et al., 2013).

3.5. Humic acid removal

The removal of natural organic matter (NOM) from water can take place through several mechanisms (Lanciné et al., 2008). These mechanisms include charge neutralization,

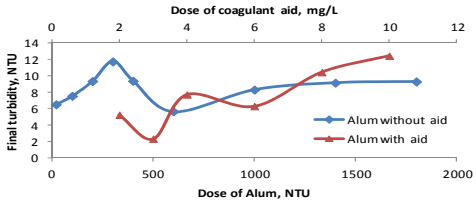

Fig.4.Effect of coagulant dose on removal of turbidity

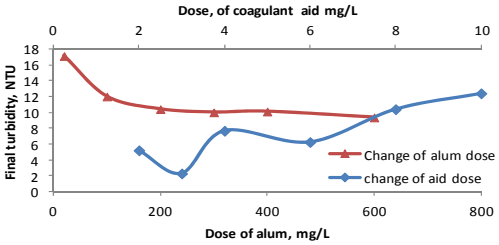

Fig.5. Optimization of alum and coagulant doses

entrapment, and adsorption. The clay minerals dispersed in natural water have the capacity to adsorb humic substances. Alum dissociates in water to produce cations. The ion exchange between the organic and clay particles due to the presence of these cations is important in this mechanism (Duan and Gregory, 2003). Hydrogen bonding and Van der Waals forces may also play a significant role, particularly if the humic molecules have large uncharged portions and sufficient flexibility to come into contact with the surface of clay particles (American Water Works Association, AWWA, 1979).

To investigate the effect of the coagulant aid for the removal of humic acid, two experiments were performed. In the first one, the dose of alum was kept constant and the dose of COL was changed, and in the second one, the dose of COL was kept constant with varied doses of alum. It is clear from Figures 6A and 6B that the removal of humic acid is closely related to the removal of turbidity. This may be due to the adsorption of humic substances on the surface of kaolin and its collective flocculation. It can be noticed that the TOC value representing humic acid and residual COL after flocculation reached a zero value at doses greater than 400 mg/L of the coagulant and for doses of 3 and 5 mg/L of the coagulant aid. These doses are considered as the optimum doses. The increase in TOC as shown in Figure 6A, after reaching the zero TOC value, is attributed to the residual coagulant aid.

3.6. Effect of Coagulant Aid on Flocculation and Settling Times

Two sets of beakers containing the turbid water were used. To the first set, the optimum doses of alum were added to each beaker, and to the second set, optimum doses of both alum and coagulant aid were added.

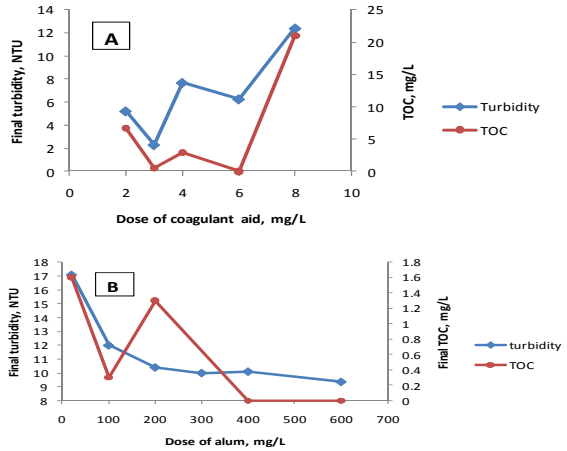

Fig.6. Optimization of alum and coagulant doses for total organic carbon (TOC) removal

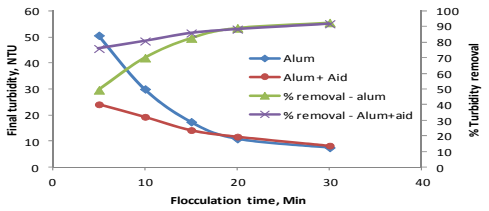

Fig.7. Effect of coagulant aid on the flocculation time

As illustrated in Figure 7, a clear difference can be observed at the initial mixing stages. The solutions containing the coagulant aid were destabilized very rapidly. Within 5 min of slow mixing followed by sedimentation, the turbidity of water treated with the coagulant aid decreased to 75% of its initial value compared to 50% in case of water treated with alum alone.

An important difference between the two systems was the size of the formed flocs. As illustrated in Figure 8, in the case of water treated with coagulant aid, large flocs were observed within 5 min, though they had irregular shapes.

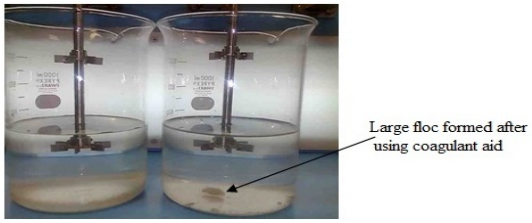

Fig.8. Effect of coagulant aid on the flocs shape and size; Left: water treated with alum alone, Right: water treated with alum + coagulant aid

The effect of coagulant aid on sedimentation time was analyzed at different time intervals using optimum doses of alum alone and alum combined with the coagulation aid. The test was performed similarly to the previous one with a slight difference; the slow mixing was continued for 20 min followed by sampling treated water at different time intervals during the settling process. Figure 9 illustrates the settling rate for both the tested systems. Alum showed a much faster sedimentation rate when used alone. A sedimentation time of 40 min was found to remove 92% of flocs while alum with coagulant aid took 70 min to achieve this value. These observations may be attributed to the shape of the flocs. The flocs formed when using alum alone were found to be homogeneous and spherical in shape that leads to rapid sedimentation rate, while flocs formed when using alum and coagulant aid were irregular in shape, which resulted in the slow rate of stirring. The flocs formed by using the coagulant aid had probably lower density and gelatinous nature compared to the flocs formed by alum that may lead to reduced settling rate. However, the water produced from the filter bed after sedimentation showed better quality for systems that used the coagulant aid. The residual turbidity in such systems was 0.26 NTU compared to 0.632 NTU when using the alum alone. This can be attributed to the same reason, and the irregular flocs were captured by the filter media much better than the spherical ones. Usually, the physical characteristics of the flocs are important in determining their removal efficiency. For example, large compact flocs have a high settling rate (Wilen et al., 2003), while large and porous flocs aid filtration due to their low permeability (Bushell et al., 2002).

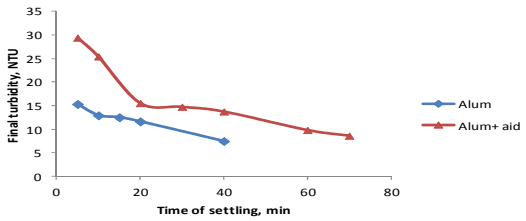

Fig.9. Effect of coagulant aid on the settling time

4. CONCLUSION

The conducted experiments confirmed the significant use of this novel coagulant aid, namely COL. The results showed that COL has the ability to reduce the dose of the primary coagulant (alum) and the residual turbidity to almost the half. The coagulant dose was reduced from 600 mg/L to 300 mg/L while the residual turbidity was reduced from 5.63 to 2.3 NTU when using the coagulant aid. COL was found to increase the initial rate of flocculation process, but it did not enhance the settling rate of the formed flocs. However, the water produced after filtration had much better quality compared to that produced without the use of coagulant aid (0.26 NTU and 0.632 NTU, respectively). The comparison between all these results indicated that COL can be used as a coagulant aid.

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مادة بيو مجمعة جديدة مساعدة لمعالجة المياه ذات العكارة العالية وتحتوي على الأحماض الدبالية

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الملخص:

يتم إنتاج المياه الصالحة للشرب من معظم مصادر المياه عادة عن طريق التخثر وذلك لإزالة العكارة. و تستخدم الشبة بشكل واسع لهذا الغرض. ومع ذلك، هناك قلق حول المخاطر المرتبطة بالشبة مثل مرض الزهايمر. ونتيجة لذلك، كان هناك اهتمام كبير في تطوير مواد تخثر طبيعية من أجل تقليل جرعة الشبة. تهدف هذه الدراسة إلى تقييم استخدام سيقان أحد انواع الخضروات (الملوخية) و التي تعتبر كمخلف زراعي لهذا الغرض. و هذا النوع من الخضار يزرع بوفرة في أفريقيا والشرق الأوسط. و الميزة الهامة لهذا المخلف الزراعي انه لا يحتاج إلى أي معالجة كيميائية. أجريت الاختبارات لتقييم الجرعة المثلى والشروط المطلوبة لتحقيق الإزالة المثلى لكل من التعكر والاحماض الدبالية من المياه. وبناء على نتائج الاختبار فهذه المادة تساعد على تخثر الماء بكفاءة كبيرة. اثبتت النتائج المعملية قدرة هذه المادة على تقليل جرعة الشبة من 600 ملغم/لتر إلى 300 ملغم/لتر و تقليل العكارة المتبقية من 5.63 الى 2.3 وحدة عكارة (NTU). خفضت هذه المادة تركيز الكربون العضوي الكلي (TOC) الى مستوى الصفر، كما انها ساعدت على ارتفاع سرعة التخثر.