

# Optimization of flocculation conditions of low concentrated clay suspensions using prepared and commercial nonionic flocculants

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The flocculation process of clay suspensions using both commercial and prepared nonionic polyacrylamide flocculants has been studied and partially optimized. Factors such as clay concentration, pH of the medium and temperature of clay suspension have been studied. Also, the effect of the aqueous medium on the flocculation efficiency has been investigated. Finally, the optimum dose for different clay concentrations in wide range of pH has been determined.

تم دراسة بعض العوامل التي تؤثر على عملية تجميع الطفلة المعلقة باستخدام مواد مجعته غير مشحونة إحداهما تجاريه والأخرى محضره معمليا . وتلك العوامل هي تركيز الطفلة في المعلق و درجة الحموضة و درجة الحرارة . أيضا تم دراسة تأثير تغير وسط المعلق على كفاءة التجميع . و في النهاية تم تحديد كمية المادة المجمع المثلئ لتركيزات مختلفة من الطفلة المعلقة على مدى واسع لدرجة حموضة كل تركيز .

**Keywords :** Flocculation, Nonionic flocculant, Clay suspension, Jar test, Wastewater

## 1. Introduction

Removal of kaolinite turbidity from the effluents before discharging into the environment is a problem faced by the clay processing industry. Some researchers [1] quote extensive application of kaolinite suspensions as a model for natural turbidity of raw surface water in Jar Test studies of coagulation and flocculation in the laboratory. Inorganic coagulants such as alum in combination with lime have been conventionally used for the removal of clay particles in the effluents from clay processing industry as well as from raw water. The sludge formed from such treatment poses disposal problems because of its aluminium content and tend to accumulate in the environment. Stauber et al. [2] have reported that although not conclusively proved, increasing concern about the residual aluminium, which may be present in water as a result of alum treatment, is being expressed by the public in connection with Alzheimer's disease. Thus, it has become necessary to develop harmless, more efficient and environment friendly flocculants for removal of turbidity in surface waters and effluents [3].

The most frequently used flocculants are the high molecular weight water-soluble polymers that are sufficiently large to bridge between particles. These polymers derived from the acrylamide monomer [4]. The nonionic homopolymer (100% acrylamide) is an effective flocculants; however, its activity can be enhanced by copolymerization with other monomers. This can introduce functional groups that have a high affinity for a particular mineral phase (e.g. hydroxamate for the iron minerals within bauxite residue), or simply provide charged groups that allow the polymer to take on an extended conformation in solution (e.g. carboxylate or sulphonate) [5].

The interactions of the polymers with the solids are dependent on many factors, which are for the most part interactive. The type of water that interacts with solids and polymer, its temperature, and pH. Mixing, shearing by flow, and other mechanical influences in a processing operation are also important. Flocculation becomes a resultant of all these parameters. In particular, we were interested in the effect of varying the suspension pH, temperature, and the water nature of the suspended waste. This article describes the

interactive influence of flocculant dosages, waste solid concentration, pH, temperature on the flocculation of a synthetic clay suspension by two nonionic polyacrylamide flocculants, one of them is the previously laboratory prepared, and the other is the available commercial (Magnafloc7025) one. A comparison between the two flocculants on the optimum flocculant dosage and flocculation efficiency was made at various flocculation conditions. These flocculation capabilities are monitored through the measurements of the residual turbidity after 10min and then the percentage turbidity removal was calculated.

## 2. Materials and experiments

### 2.1. Materials

1. Acrylamide was supplied by Fluka chemicals Co. Ltd. (Switzerland), assay  $\geq 99\%$ . Potassium persulphate [ $K_2S_2O_8$ ] supplied by S.D.S. FINE-CHEM Ltd. (England), min assay 98%, chloride not more than 0.04%.

2. Clay powder, Egyptian highly refined Ball clay (smectite- vermiculite) powder of very fine particle size, used mainly for manufacturing refractory bricks and mortar, was obtained directly from Alexandria for Refractories Company. The physical and chemical characteristics of the material (data provided by the manufacturer) are as shown in table 1.

3. MAGNAFLOC7025, is a high molecular weight nonionic polyacrylamide flocculants (Ciba Specialty Chemicals, U.K) are supplied as a free flowing granular powder, its properties as follow: (physical form: white granular powder, particle size: 98%  $< 1000\mu m$ , bulk density:  $0.8g/cm^3$ , pH of 1% solution at  $25^\circ C$ : 5.5, viscosity at  $25^\circ C$  of 0.5% solution at shear rate 5.11: 50 cP, relative viscosity at  $25^\circ C$ : 4.1).

### 2.2. Preparation of polyacrylamide

The preparation conditions were 6% monomer concentration, 0.001M potassium persulphate, and polymerization time 55min at  $50^\circ C$  using solution polymerization technique. The yield of acrylamide monomer converted to polyacrylamide flocculant was 71.5%. The

Table 1  
Characterization of clay powder

Particles size	- 325 mesh
Moisture	1% maximum
Silica	51.4%
Alumina	32%
Ferric oxide	2.6%
Titania	2%
Calcium oxide	0.3%
Sodium oxide	0.1%
Magnesium oxide	0.12%
Potassium oxide	0.08%
Loss on ignition	11%.

produced flocculant has relative viscosity at  $25^\circ C$ ; 45.

The procedure of polymerization is conducting as follow:

1. Dissolve the monomer acrylamide in distilled water to prepare acrylamide solution of the desired concentration and then the solution is purged with nitrogen gas for 30min at the selected temperature.
2. Predetermined weight of potassium persulphate is added to the monomer solution with continuous bubbling of nitrogen gas for 10 minutes.
3. Temperature of the reaction medium is adjusted to start the polymerization reaction.
4. After 30 minutes, the stirring is stopped but the nitrogen gas continues to pass through the polymerization vessel atmosphere.
5. After completion of the desired polymerization time the reaction medium is diluted with distilled water to give the selected flocculant concentration (0.5%) and the medium stirred gently with mechanical stirring until a homogeneous solution is obtained.

### 2.3. Preparation of clay suspension

Clay suspensions were prepared from clay powder immediately before each set of experiments, through mixing accurate weight of clay powder with certain volume of distilled water.

### 2.4. Preparation of flocculant solution

A concentrated flocculant solution was prepared by first wetting the surface of the flocculant (0.5g) with 3ml of ethanol, and complete until 100ml with distilled water. The sample then was stirred for 2 hours to have a

homogeneous 0.5% (w/v) solution that stored as stock solution.

### 2.5. Jar test

1. Clay suspensions with different initial concentrations; 0.1-1%; were prepared by weighing accurately the required amount of clay and mix it with 800 ml of tap or distilled water.

2. The suspension was prepared at the desired initial pH using (0.1M HCL & 0.1M Na<sub>2</sub>CO<sub>3</sub>). pH variation was 2-11. pH monitored using pH-meter (Denver Instrument Co., U.S.A.).

3. To maintain clay suspension at constant temperature for studying the effect of temperature change in flocculation process *Mechanical convection oven* (Carbolite Co., England) is used for this purpose.

4. The previously prepared suspension was put in each of five 1L beakers and stir for two minutes at 190 rpm using Laboratory Flocculator (Flocumatic 6PLAZAS/samples) model-3000914 (J.P. Selecta Co.), which consists of six-spindle multiple stirrer units with stainless steel paddles, in order to assure complete dispersion of clay in water. Afterwards, a settling time of 10 min was allowed.

5. At the end of the settling period, the sample for turbidity measurement was withdrawn using automatic pipette from a height of 3 cm below the surface of each beaker. These samples measured using 2100 p Turbidimeter (Hach Co.) calibrated using standard suspensions, which represent the initial turbidity ( $T_0$ ) for each clay concentration.

6. The required volume of 0.5% polyacrylamide solution was added to each test suspension.

7. Immediately after the addition of the flocculants, the suspension was stirred at a uniform speed of 75 rpm for 2 min., this was followed by a slow stirring at 25 rpm for 5 min.

8. The stirrer was then switched off and the flocs allowed settling undisturbed for 10 min.

9. The residual turbidity ( $T$ ) of the supernatant liquid was measured as described previously.

10. The percentage removal of turbidity using polyacrylamide flocculants was calculated according to the following equation

$$\% \text{Turbidity removal} = [(T_0 - T) / T_0] * 100 \quad (1)$$

11. These procedures were repeated using 0.5% commercial Magnafloc7025 flocculants solution instead of the prepared polyacrylamide flocculants.

12. Flocculation efficiency using both flocculants (prepared & commercial) was compared.

## 3. Results and discussion

The aggregation of clay particles suspended in the distilled water, in both the presence and absence of the two nonionic polymers was investigated for different clay concentrations at various pH suspensions. This study was carried out using distilled water in order to avoid the agglomeration effect of the salts present in tap water, which may interfere with the flocculation effect of the nonionic flocculants.

### 3.1. Effect of clay concentration & suspension pH

The precipitation of clay suspensions without using any flocculant is illustrated in table 2 for the studied clay concentrations at various pH.

It is clear from this table that for each clay concentration as pH is increased from 2 to 11 the initial clay turbidity increased. This may be due to the fact of the negative natural surface of clay particles [6]. Increasing pH results in increasing the negative charge on the edges of clay layers and when two charged surfaces are brought together their diffuse layers overlap, an electrical force would exist between the two surfaces and for surfaces with similar

Table 2  
Effect of both clay concentration & pH on the supernatant turbidity

pH	Clay concentration (%)				
	0.1	0.25	0.5	0.75	1
2	173	166	112	110	105
3	297	251	188	164	109
7	702	1000	1424	1562	1696
9	948	2008	3972	5520	7950
11	1000	2890	5550	7350	9780

charges [7] repulsive force exists. The magnitude of this repulsive force is related to the magnitude of charge on the particles, as this charge increased the repulsive force is increased; this is by its role increases the stability of clay suspension. So particles can approach each other most closely when they are not highly charged because increasing the surface charge of particles should cause stabilization of a suspension. This indeed what happens in the absence of polymer a suspension let to settle at pH 2 was much less turbid than one let to settle at a higher pH. These results are in agreement with Chandra W.A., et al. [8]; they studied the flocculation of kaolin as a function of pH.

Also it can be deduced from this table that for acidic media (pH 2,3) the increase in clay concentration leads to a decrease in initial turbidity. This is returned to the aggregation rate is proportional to the square of the concentration of the particles [9]. Also, as mentioned above that particles can approach each other most closely and settled when they are not highly charged at low pH so as clay concentration is increased the aggregation of clay particles become faster and after 10min the initial turbidity decreased.

In contrast to the acidic media the neutral and alkaline media (pH 7-11) show an increase in initial turbidity as the clay concentration is increased. This is due to increase the mutual repulsive forces between clay particles. Further as clay concentration is increased these repulsive forces become higher due to the increase of clay density in suspension. These forces not only prevent the particles from coming together but it also retard settlement by keeping the particles in constant motion [10]. So the initial turbidity after 10min has increased as clay concentration increase.

### 3.2. Effect of flocculant dosages

The relationship between flocculant dose and the percentage turbidity removal for both nonionic flocculants at acidic and neutral media (pH 2, 3, 7) increased then decreased as flocculant dose increased as illustrated in fig. 1 that represent this effect for 0.5% clay suspension at pH 3.

This is in agreement with the expected result because the flocculation in a given system cannot be increased beyond a certain optimum dosage of polyelectrolytes, and further additions result in decreased efficiency [9]. This result reveals that the most effective flocculation is obtained using the optimum flocculant concentration. This is because the larger amount of flocculant in the suspension causes a larger amount of suspended particle to aggregate and settle. However, an over optimal amount of flocculant in the suspension would cause the aggregated particles to re-disperse in the suspension and would also disturb the particle settling [11].

On the other hand concerning the alkaline media (pH 9, 11) the second flocculation behavior shows an increase in the percentage turbidity removal as flocculant dose is increased then on further increase in the flocculant dose, percentage turbidity removal becomes constant or increased slightly for all studied clay concentrations (0.1-1%) as shown in fig. 2. This may mean that the flocculant did not arrive to the over dosing so it cannot cause re-dispersion of the aggregated particles (up to 4 ml dosage added). This may be attributed to what has been previously mentioned that the increase in pH increases the charge density on clay particles and this in turn increases the dispersion of clay particles in suspension and flocculation become more difficult. This is due to the increase in clay surface charge keeps the particles apart and bridging become less frequent as more polymers is adsorbed on single particles [9]. Thus it can be expected

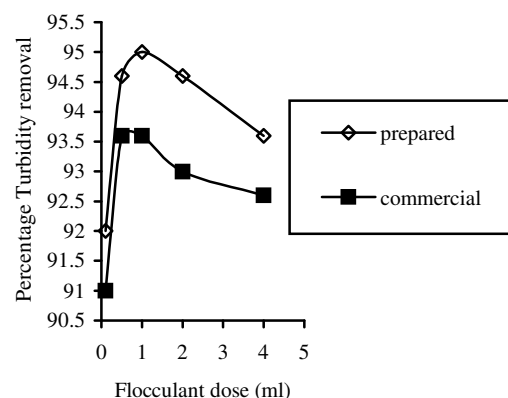


Fig. 1. Effect of flocculant dose on %turbidity removal of 0.5% clay suspension at pH 3.

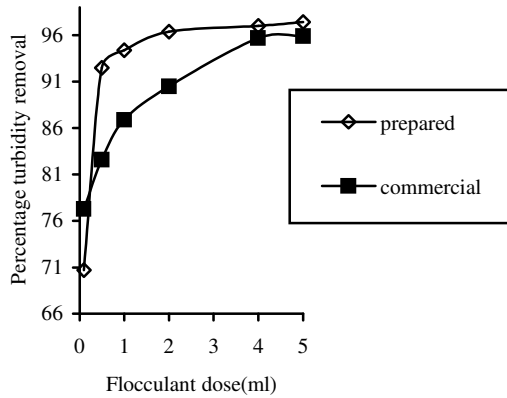


Fig. 2. Effect of flocculant dose on % turbidity removal of 0.5% clay suspension at pH 9.

that there is no excess flocculant present in clay suspension to cause re-dispersion of the flocculated particles.

### 3.3. Optimization of flocculant dosages

From the previous flocculation study of the different clay suspension concentrations at various pHs, the optimum flocculant dosages and the corresponding optimum percentage turbidity removal were determined for each clay concentration at the various studied pH for the two nonionic flocculants (prepared & commercial).

Concerning to the prepared flocculant, it is observed from fig. 3. that at the same clay concentration as the suspension pH increased from 2 to 11 the optimum flocculant dose required increased from 0.5ml to 2ml. Moreover for the acidic and neutral (pH 2, 3, 7) as clay concentration increased the flocculant dose required decreased. This is may due to as pH decreased the clay particles charge decreased and the distance between clay particles become narrow and as clay concentration increased this distance become narrower due to the increase in clay density in suspension so the flocculant dose required for bridging decreased and the rate of aggregation increased as clay concentration increased. This observation does not noticed in case of alkaline media (pH 9, 11) because there is repulsion between clay particles.

On the other hand fig. 4 represent the optimum dose required when commercial Magnafloc7025 used. It is noticed that the

trend in this case is similar to the prepared flocculant case, where it is clear that optimum flocculant dose required increased from 0.5ml to 4ml as the suspension pH increased. Also, similarly to the prepared flocculant at acidic and neutral (pH 2, 3, 7) as clay concentration increased the flocculant dose required decreased. However It is noticed that at the acidic and neutral media (pH 2,3,7) the optimum dose ranged from 0.5 to 2ml when commercial flocculant used and from 0.5 to 1ml in case of prepared flocculant for the different studied clay concentration. And at alkaline media (pH 9,11) the optimum dose is 4ml in commercial flocculant case compared with 2ml required for prepared flocculant.

The corresponding optimum percentage turbidity removal yielded from using the previous optimum flocculant dosages for both prepared and commercial Magnafloc 7025 flocculants are investigated in fig. 5.

It is deduced from this figure, at the neutral and acidic media (pH 2,3,7) that the increase in clay concentration shows an increase in optimum percentage turbidity removal. This result support the observation of Ravi D., and Sivasankara Pillai V.N. as they observed that at higher initial concentrations of suspended solids, the flocs appear in a shorter time and grow to a larger size [3]. This observation is reversed in case of alkaline media (pH 9, 11), where it is noticed a decrease in optimum percentage turbidity removal as clay concentration increased. This is may returned to the increase in clay concentration increases the mutual repulsive force between clay particles.

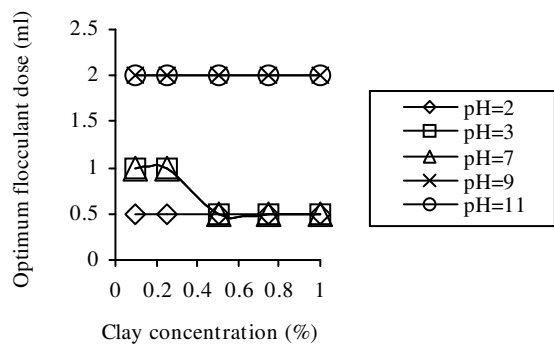


Fig. 3. Optimization of prepared flocculant dosage for different pH using different clay concentrations.

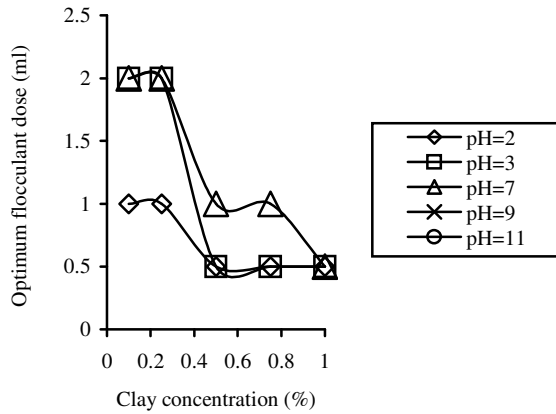


Fig. 4. Optimization of commercial Magnafloc7025 flocculant dosage for different pH using different clay concentrations.

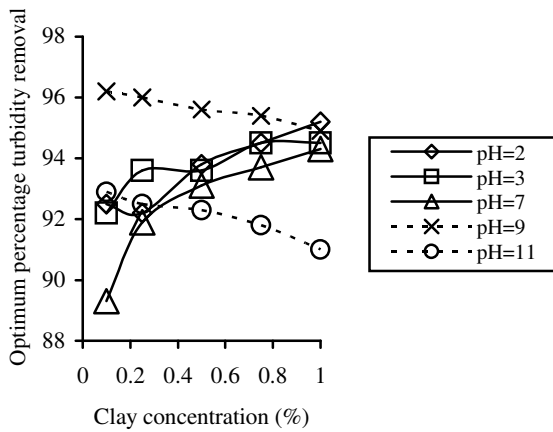
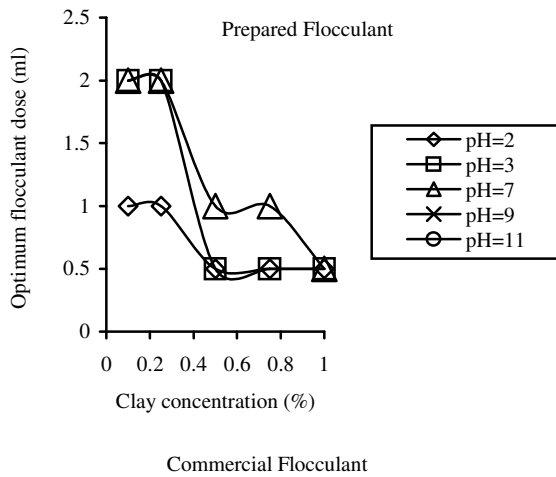


Fig. 5. The yielded optimum % turbidity removal from using the optimum dosages for both prepared & commercial for the different pH using different clay concentrations.

### 3.4. Effect of aqueous medium

In order to study the effect of salts presence in the tap water on the flocculation process, the effect of suspension media on agglomeration clay particles with and without using flocculant are studied using both distilled and tap waters.

Fig. 6 represent the residual turbidity of clay suspension settled without using flocculant at the two different aqueous media. It can be seen that clay suspensions settled in distilled water show higher residual turbidity than the other at tap water for all clay concentrations. This is may be due to the effect of presence of some constituents in tap water that are not present in distilled water [3], because tap water supplied from river is subjected to conventional treatment (coagulation using alum and lime, settling, filtration and chlorination) before supply. Distilled water produced through the distillation of this tap water to remove all ions present. These constituents will assist the aggregation of clay particles in the suspension by reducing the net surface charge of the particles in the suspension. As the net surface charge is decreased the thickness of the diffuse layer surrounding the particles is reduced and the particles become more adjacent and agglomerated together then settled faster than discrete ones [10]. So the residual turbidity after 10min at tap water media decreased compared to distilled water.

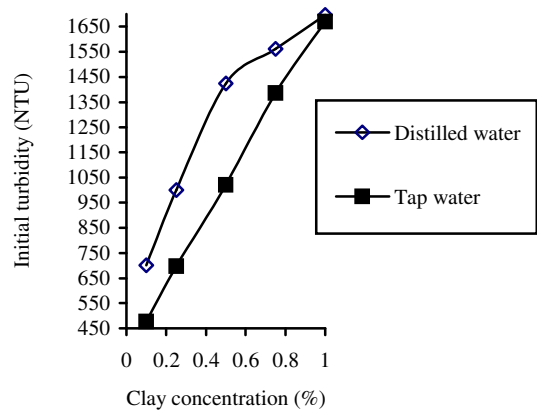


Fig. 6. Effect of suspension medium on the supernatant turbidity for different clay concentrations.

On the other hand figs. 7 and 8 represent the effect of both prepared and commercial flocculant dosages on the percentage turbidity removal for the different studied clay concentrations.

It is observed from these figures that the flocculation behavior for both nonionic flocculants in tap water is similar to their behaviors in the distilled water, i.e. the percentage turbidity removal increased then decreased as flocculant dose increased for all clay concentrations.

The optimum flocculant dosages and the yielded optimum percentage turbidity removal are determined from the previous figures for each aqueous media. The optimum flocculant dosages for the different aqueous media are tabulated in table 3 and the corresponded percentage turbidity removal investigated in fig. 9.

From this table it is clear that for both nonionic flocculants the optimum flocculant dose required at tap water is smaller than the one required at distilled water. Also on examining the percentage turbidity removal from fig. 9, it is noticed for both nonionic flocculants that the optimum percentage turbidity removal yielded in tap water is higher than that yielded on using distilled water. From these results it is concluded that in spite of using smaller flocculant dosages in case of using tap water than in distilled water case, the performance of each flocculant in turbidity removal is more efficient due to the presence of minute quantities of ions, which are not present in the distilled water.

### 3.5. Effect of suspension temperature

Since the previous study indicated that the flocculation efficiency is higher in case of tap water than using distilled water. So the effect of suspension temperature on the flocculation behavior of both nonionic flocculants was studied using tap water and the predetermined optimum flocculant dosages are used for the different clay concentrations studied.

It is noticed from fig. 10 that for both nonionic flocculant as the temperature increased the residual turbidity decreased for each clay concentration. consequently the flocculation performance increased as tem-

perature increased. This is due to the rate of diffusion of flocculants and the rate of collision of particles increases with a rise in temperature [9]. Furthermore an increase in turbidity removal is noticed as clay concentration is increased. This is may be due to the increase of aggregation rate as the initial clay concentration is increased [9].

On the other hand by comparing the effect of prepared flocculant on the clarity of the supernatant with the commercial one, it is noticed that prepared flocculant shows a higher efficiency in reducing the supernatant turbidity than the commercial one at all studied temperature.

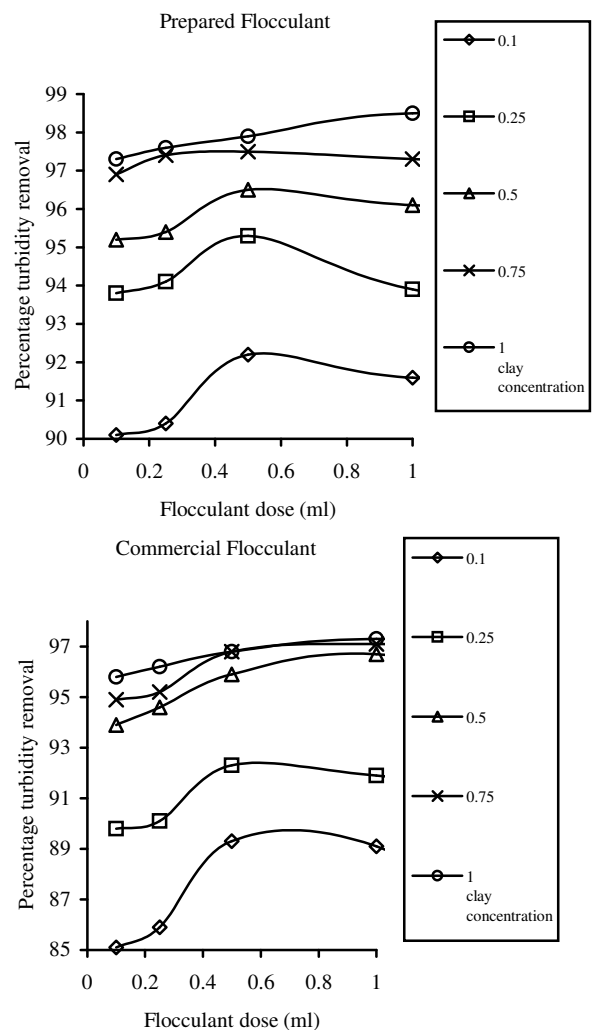


Fig. 7. Effect of flocculant dose on %Turbidity removal for different clay concentrations using distilled water.

Table 3  
Effect of aqueous media on the optimum flocculant dose required for both nonionic flocculants

Clay concentration (%)		0.1	0.25	0.5	0.75	1
		Optimum flocculant dose (ml)				
Prepared flocculant	Tap water	0.5	0.5	0.5	0.5	0.5
	<u>Distilled water</u>	1	1	0.5	0.5	0.5
Commercial flocculant	Tap water	0.5	0.5	0.5	0.5	0.5
	Distilled water	2	2	1	1	1

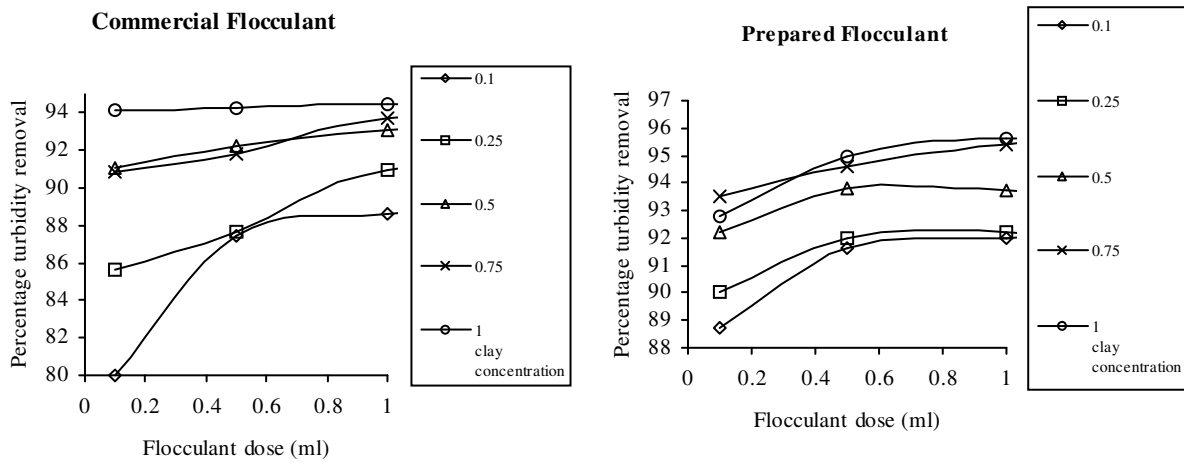


Fig. 8. Effect of flocculant dose on %Turbidity removal for different clay concentrations using tap water.

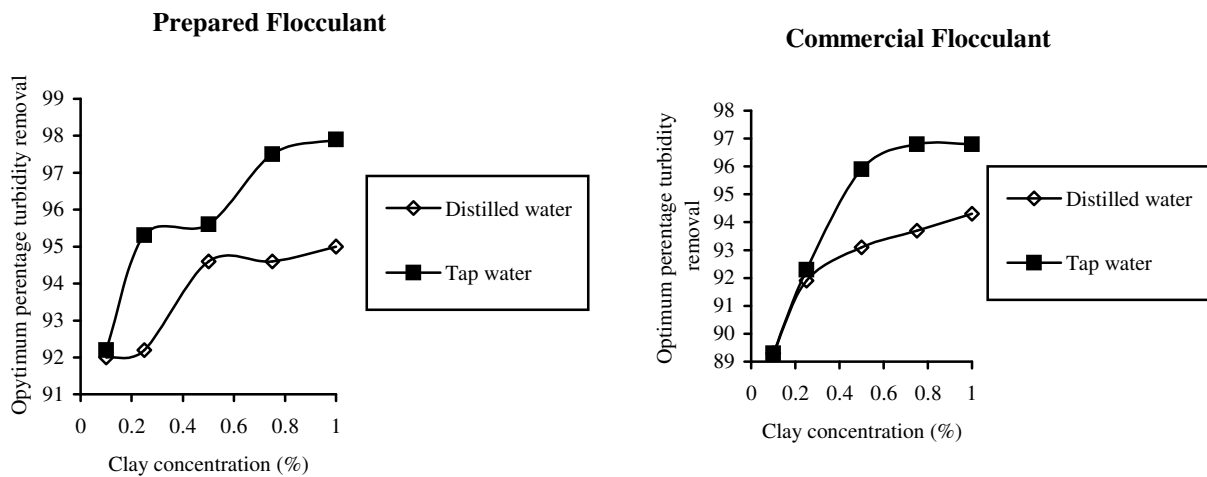


Fig. 9. Effect of aqueous medium on %Turbidity removal using optimum dose of the two nonionic flocculants (prepared & commercial).



3.6. Comparison the flocculation capabilities among prepared and commercial Magnafloc7025 flocculants

Fig. 11 represents one of the laboratory experiments. It is clear from this figure that the blank suspension (settled without flocculant) shows a very cloudy suspension compared to the other two flocculated suspensions. Also when commercial flocculant used the residual supernatant showed a higher turbidity than the prepared flocculant. Further more both figs. 1 and 2 illustrate that prepared flocculant give higher percentage turbidity removal than the commercial one at

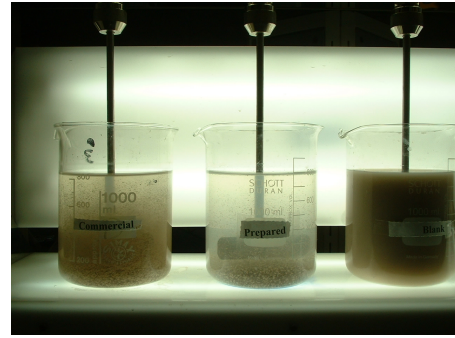


Fig.11. Flocculation effect of commercial and prepared flocculants.

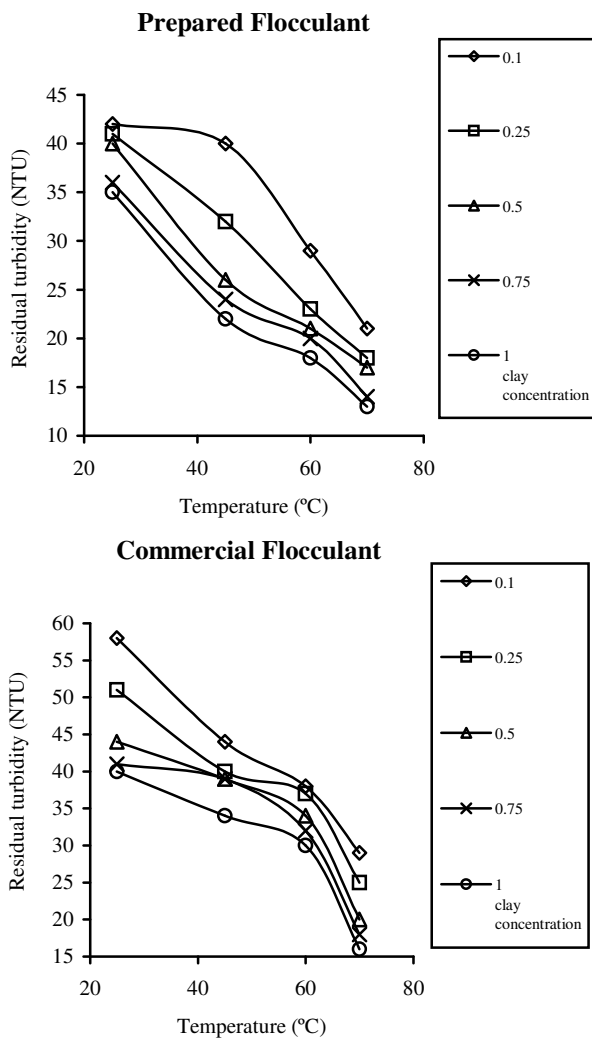


Fig. 10. Effect of temperature on residual turbidity for different clay concentrations using tap water and optimum flocculant dosages.

the same flocculant dosages. Moreover figs. 3, 4, 5 and 9 indicates that the prepared flocculant is most economical than the commercial Magnafloc7025 in treatment clay waste especially when the optimum flocculant dose of the prepared flocculant required to give equal or much higher turbidity removal is smaller than the commercial one required even this waste suspended in distilled or tap water. All these results are verified that the flocculation efficiency of the prepared flocculant is higher than the commercial one. This is returned to the larger relative viscosity of prepared flocculant (45) than the commercial one (4.1), which indicate that the molecular weight of prepared flocculant is much higher than the commercial one.

4. Conclusion

From the previous remarkable results, it is concluded that the acidic media cause agglomeration of dispersed clay particles inconsistency to the alkaline media, which increase the dispersion of clay particles in suspensions. So for both nonionic flocculants the relationship between flocculant dose and the percentage turbidity removal follows two different attitudes. Such results indicate that we have two different flocculation behaviors, the first one in acidic and neutral media (pH 2, 3, 7) where the percentage turbidity removal increased then decreased as flocculant dose increased, while in the second one in alkaline medium (pH 9, 11), it is noticed continuous increase in the percentage turbidity removal as flocculant dose increased in the range of study. These results reveals that a very low flocculant dose required in acidic and

neutral media because presence of larger amount of flocculants causes re-dispersion of flocculated particles and over dosing take place. However this phenomenon does not occur in alkaline medium. Moreover, in acidic and neutral media the increase in clay concentration leads to increase in percentage turbidity removal but in alkaline media this cause decrease in the percentage turbidity removal. The best flocculation results in acidic and neutral media were obtained when the flocculant dosages ranged between 0.5 to 2ml when commercial flocculant used and between 0.5 to 1ml in case of prepared flocculant for the different studied clay concentrations. The optimum yielded percentage turbidity removal from using these optimum doses extended from 92 to 96 using prepared flocculant and 89 to 95 in case of commercial one. However, at alkaline media the best dose is 4ml in commercial flocculant case compared with 2ml required for prepared flocculant. The corresponding yielded percentage turbidity removal gradient from 97 to 93 using prepared flocculant and from 96 to 91 when commercial flocculant used. However these optimum doses decreased to 0.5ml for the two nonionic flocculants for all studied clay concentrations for clay suspended in tap water. Due to the presence of trace quantities of ions in tap water that is effectively reduce the clay turbidity and improve the flocculation efficiency than in distilled water case. The increase in suspension temperature improves the flocculation efficiency using any one of the studied non-ionic flocculant for each clay concentration. Also the increase in the clay concentration followed by decrease in the residual turbidity. Comparing the flocculation performance of the prepared flocculant with the identical Magnafloc7025 flocculant, it is deduced that the laboratory prepared flocculant is most economical than the compared commercial Magnafloc7025 flocculant. Since smaller doses of prepared flocculant show better performance under almost all conditions studied.

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