Low Technology Water Treatment: Investigation of the Performance of Cactus Extracts as a Natural Flocculant for Flocculation of Local Clay Suspensions

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Abstract--This study aims to find news a die of purification for a wastewater reuse. Primary effluent contains organic mineral, dissolved and suspended matter (colloids). Microfiltration or ultra-filtration is adequate for producing disinfected clear water suited for different applications. However, direct filtration on membrane is limited by the fouling phenomena which leads during filtration to constant pressure, to a strong and continuous decrease of the permeate flux. On the other hand, coagulation and flocculation make it possible to removal the colloidal fraction, which plays a significant role in membrane fouling. An evaluation of fives extracts from cladode of Opuntia Ficus Indica (OFI), extracted by different solvents at high temperature was investigated, as coagulant aids with lime as coagulant. Experiments were conducted to determine optimum conditions for treating the turbid water loaded by clays from lakes in Morocco. The turbidity removal efficiency for two extracts was greater than 95% and can achieve 99% at optimal conditions of dosage and pH.

Keywords — *coagulation-flocculation, water treatment, cactus, pectins, mucilage.*

I. Introduction

The cactus Opuntia (genus Opuntia, subfamily Opuntioideae, family Cactaceae) is a xerophyte producing about 200–300 species and is mainly growing in arid and semi-arid zones. Due to their remarkable genetic variability, Opuntia plants show a high ecological adaptivity and can therefore be encountered in places of virtually all climatic conditions [1, 2].

Commercial cultivation is carried out in Italy, Spain, Mexico, Brazil, Chile, Argentina, and California [3]. Traditionally and still today, cactus plants serve as sources for fruits and vegetables, for medicinal and cosmetic purposes, as forage, building material, and as a source for natural colors. However, their use is still mainly restricted to the countries of origin [1,4,5,6]. The production of mucilages, often referred as pectin polysaccharides, is characteristic of members of the Cactaceae family [7]. Several properties are allotted to pectins as cementing agents in plant cell walls [8] or during fruit ripening [9], but they are mainly known for their roles in food processing [10] and as dietary fibre. When extracted from plants, these pectins are used as thickening agents in the food and in the pharmaceutical industries [11] and as bioflocculant in water treatment.

Bioflocculants have received considerable scientific and biotechnological attention in recent years due to their biodegradability, benign nature and lack of secondary pollution of their degradative intermediates [12, 13]. Flocculation is one of the important treatments given to the industrial effluent before discharging them into rivers to remove toxic waste. Organic polymeric flocculants have been used in water purification for several decades.

Preparation of plant originated hydrocolloids generally begins with extraction from the source using water, acid or alkaline solutions. Various studies have shown that parts of plants and extraction conditions significantly influenced the yield and physicochemical characteristics of plant polysaccharides [14, 15]. The main aim of the pectin industry is therefore to obtain water-soluble pectin preparations of high molecular weight and specified degree of methoxy-esterification (DM) and degree of amidation which are able to form gels under specified conditions [16], in addition to good pectin yields. The main factor governing the gelling mechanism of pectin is its DM. Thus, pectin with a DM > 50, called high-methoxy pectin (HMP), needs sugar (such as sucrose) at a concentration ≥ 55 wt % and acid (pH 2.0-3.5) to form gels, whereas pectin with a DM< 50, called low-methoxy pectin (LMP), needs calcium ions to form gels within a larger pH range (2.0-7.0) whether sugar is present or not [17]. In reference to these works, the present study relates to the treatment of turbid waters by coagulation-flocculation using mucilage and differents pectins extracts from leaves of cactus OFI.

II. Materials and Methods

A. *Materials*

A batch of fresh leaves of OFI, was obtained from a plantation in Berrechid, Morocco, and stored at 4◦C until mucilage and pectins extraction (period not exceeding 10 days). Harvesting of cladodes was carried out in January 2014. The chemicals used in the experiment were of analytical grade except lime.

Natural soil was used to prepare Synthetic turbid water, the samples loaded with Suspended solids are obtained from a dry lake that lies in the province of Berrechid, turbidity may be created from a wide variety of eroded materials, including clay, silt, or mineral particles from soils, or from natural organic matter created by the decay of vegetation.

B. Methods

1) Mucilage and pectins extraction

Prior to pectin extraction, the fresh cactus pads were cleaned to remove thorns and cut into small pieces (1*1 cm) with a kitchen knife. Cactus pieces were heated in water at 85°C for 20 min to inactivate enzymes and left to cool to ambient temperature; neutralized to pH 7.5 from initial pH 4.0 in order to induce de-esterification of methoxyl groups and filtered through a cloth filter to extract as much mucilage as possible [18]. Pectic polysaccharides were sequentially extracted from residue (R1) by water (2 × 2 hours at 85 °C) [19], then the solid residue R2 was suspended in a NaOH 50 mM sodium hexametaphosphate 7.5 g/ L solution (pH 12.0)[18, 20] stirred continuously (2*1 hours at 85°C), then the residue R3 were extracted with HCl 0.05 N (2 × 2 hours at 85 °C) [21] and finally the residue R4 were extracted with aqueous solution of 0.5% oxalate [22](0.25% oxalic acid + 0.25% ammonium oxalate) (2 × 2 hours at 85 °C). The extraction scheme is given in Fig. 1.

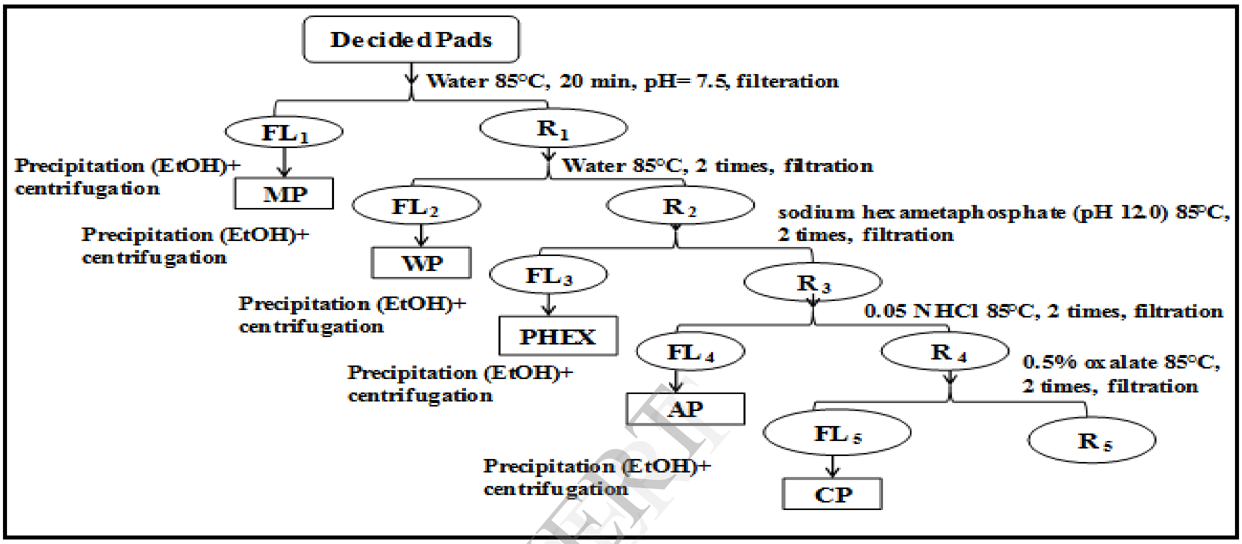

Fig. 1. Extraction of mucilage and pectic polysaccharides from cactus cladode. Where: MP: Mucilage Pads, AP: Acid-soluble Pectin, WP: Water-soluble Pectin, CP: Chelating-soluble Pectin, PHEX: Hexametaphosphate-soluble Pectin, R: Residue and FL: Filtrating liquid.

All extracts were separated from the residues by filtering through a nylon cloth [18] , and the pectin was precipitated by centrifugation (30 min, 4500 rpm) with 2 volumes of ethanol resulting in a Mucilage Pads (MP), Water-Soluble Pectin (WP), sodium hexametaphosphate-Soluble Pectin (PHEX), acid-soluble Pectin (AP) and Chelating-Soluble Pectin (CP), respectively.

2) Pectin and mucilage purification

Mucilage and Pectin coagulates were washed several times with 50, 70, 85% ethanol and finally with pure ethanol before drying at room temperature. The extraction yield of pectin production was calculated by dividing the dry weight of pectin by the dry weight of starting cladodes.

3) Optimization of coagulation-flocculation parameters

Efficiency and mechanism of coagulation-flocculation process depend on several factors, the most relevant being initial turbidity, pH, reagents (coagulant, adjuvant) dosage and type, system hydrodynamics in coagulation and flocculation stages, temperature, alkalinity [23-25]. Coagulation tests have been performed according to jar-test method, using a jar-test VELP Scientifica JLT6, equipped with six mixing posts. Coagulation step was done by fast mixing (150 rpm) for 5 minutes, while flocculation step was done for 60 minutes, at 40 rpm. All tests have been performed in 1 liter cylindrical bakers, at ambient temperature, by using stock suspensions prepared from water and soil.

The coagulants used in this work processes coagulation/flocculation, are lime (Ca(OH)2) .The flocculants used for flocculation are the mucilage and pectins extracted from cladodes. The pH was adjusted with drop wise addition of a 5% lime slurry or 0.1M sulphuric acid before adding the flocculants, to selected values between pH 2.0 and 12.4 during a second 5-min rapid stir. Samples of water, including aggregated matter, were removed from the jar at specified time intervals after stirring had ceased. Samples were always removed from exactly the same point in the jar. This point was 2 cm below the surface of the water in order to limit the influence of vertical aggregation. The optimization of the dose of the used flocculants was performed at the pH optimized to 11.5 and increasing the doses of the flocculants of mass percentage going from 0.5% to 5%. To perform optimization of pH, a known volume of 2 % biopolymer solution was added in a flocculation step to turbid water using a pipette. Lime is used widely during potable water treatment as a coagulant, to adjust coagulation pH or for stabilization of treated water.

All tests were carried out at ambient temperature. Effects of lower or higher temperatures were not studied. The residual turbidity was used as the parameter to judge the performance of the process. Data were processed using Microsoft Excel, and the curve of turbidity vs. flocculation time was plotted.

4) Preparation of turbid water samples:

About 1 kg of soil from the river located in Berrechid Morocco was thoroughly mixed with 1 l of distilled water for 4 hours and kept overnight. Resulted supernatant has been used in coagulation tests. Initial turbidity has been adjusted by adding fresh water. Turbidity has been determined by using Digital Nepheloturbidity Meter (HANNA LP2000-11). The pH was measured using a Digital pH multi-parameter (Consort C 3050). The supernatant obtained has the following properties: brown liquid, turbidity about 1000 Nephelometric Turbidity Unit (NTU) and pH=8.

III. Results and Discussion

A. *Optimization of pH*

For Dominguez et al. (2007) [26], within all the factors involved in coagulation-flocculation process, those that most affect the process efficiency are pH and coagulant dose, since influence in the hydrolysis equilibria of coagulant species. Jar test experiments were performed to assess turbidity removal as well as coagulation flocculation kinetics at various pH values. This was achieved by adding a constant flocculants dosage of 2% to samples adjusted to pH 2.0–12.4. Fig. 2 and Fig. 3 shows the effect of pH on the coagulation/flocculation process for the combination of polyelectrolyte-lime and polyelectrolyte-acid for the flocculants dose set at the pre-determined optimum value. It can be seen that for lime or acid and AP, PHEX and CP the change in turbidity with the pH is not significant. From Fig. 2C, 2E, 3A and 3C, it is seen that MP and WP produces appreciable reduction of turbidity only between pH 11.0 and 12.0. In addition, it has been mentioned by many workers that the studies for removal of turbidity were done at pH 8 and above for high turbidities [27]. The final turbidities recorded show large variations between all bioflocculants. The maximum efficiency is observed at pH 11.3 and 12.4 for MP about 98.5% removal of turbidity is achieved at this pHs, and the residual turbidity drops below 5 NTU. The flocs obtained are very coarse and settled almost completely in less than 10 min. For WP the maximum efficiency is observed at pH 11.3 and 11.5, about 99% removal of turbidity is achieved at this pH 11.5, and the residual turbidity drops below 4 NTU. The lowest residual turbidities observed for AP was 87 NTU close to pH 11.5 with a maximum removal of 86% (see Fig. 2B and Fig. 3B), 78 NTU was observed for PHEX close to Neutral pH with a maximum removal of 78% (Fig. 2D and 3D) and finally 119 NTU was observed for CP close to pH 2 about 62% as a maximum removal (Fig. 2F and 3E). From Fig. 3B, 3D and 3E the turbidity rise relative to that of blank (without added flocculants Fig. 2A) and can achieve -250% (CP). In all cases at low pH values the performances are lower and they increase to maximum values for a determined pH or pH range, after which there is another fall off in elimination efficiency at high pH.

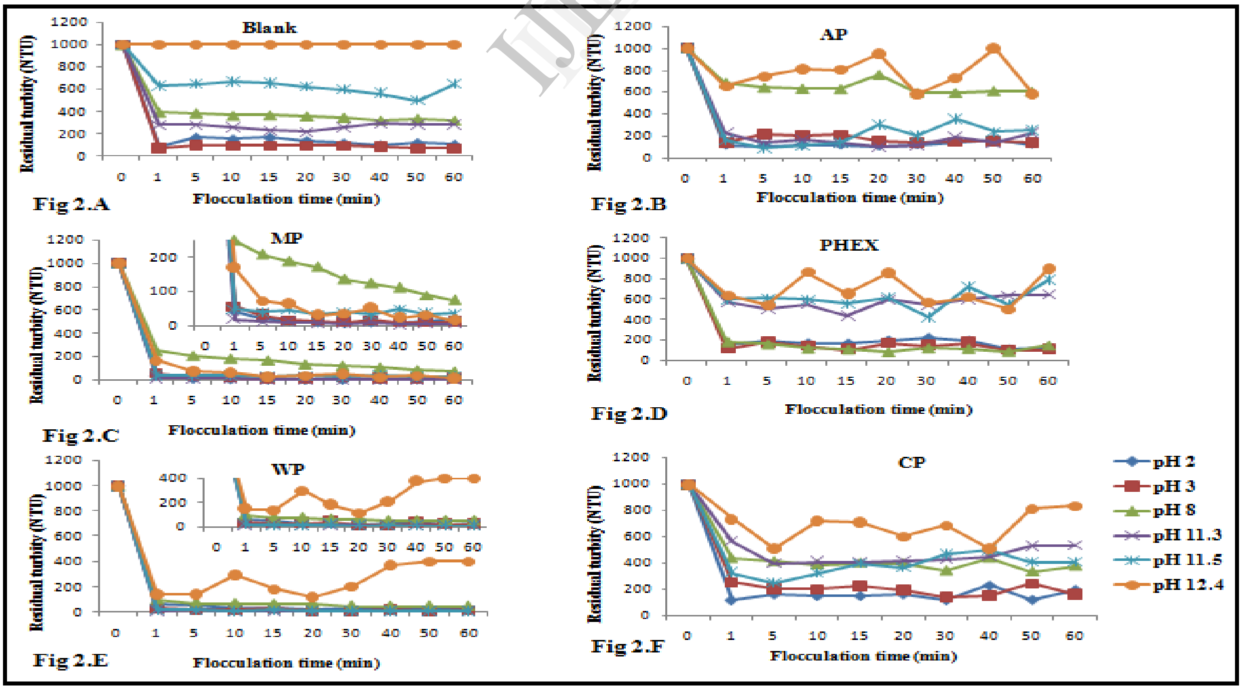

Fig. 2 . The effect of pH on the final turbidity using doses of cactus extracts of 2% (initial turbidity 1000 NTU). in fig 2.A we show the residual turbidity at differentd pH without using flocculants and in Fig 2.B, 2.C, 2.D, 2.E and 2.F plots the residual turbidity using AP, MP, PHEX, WP and CP flocculants, respectively.

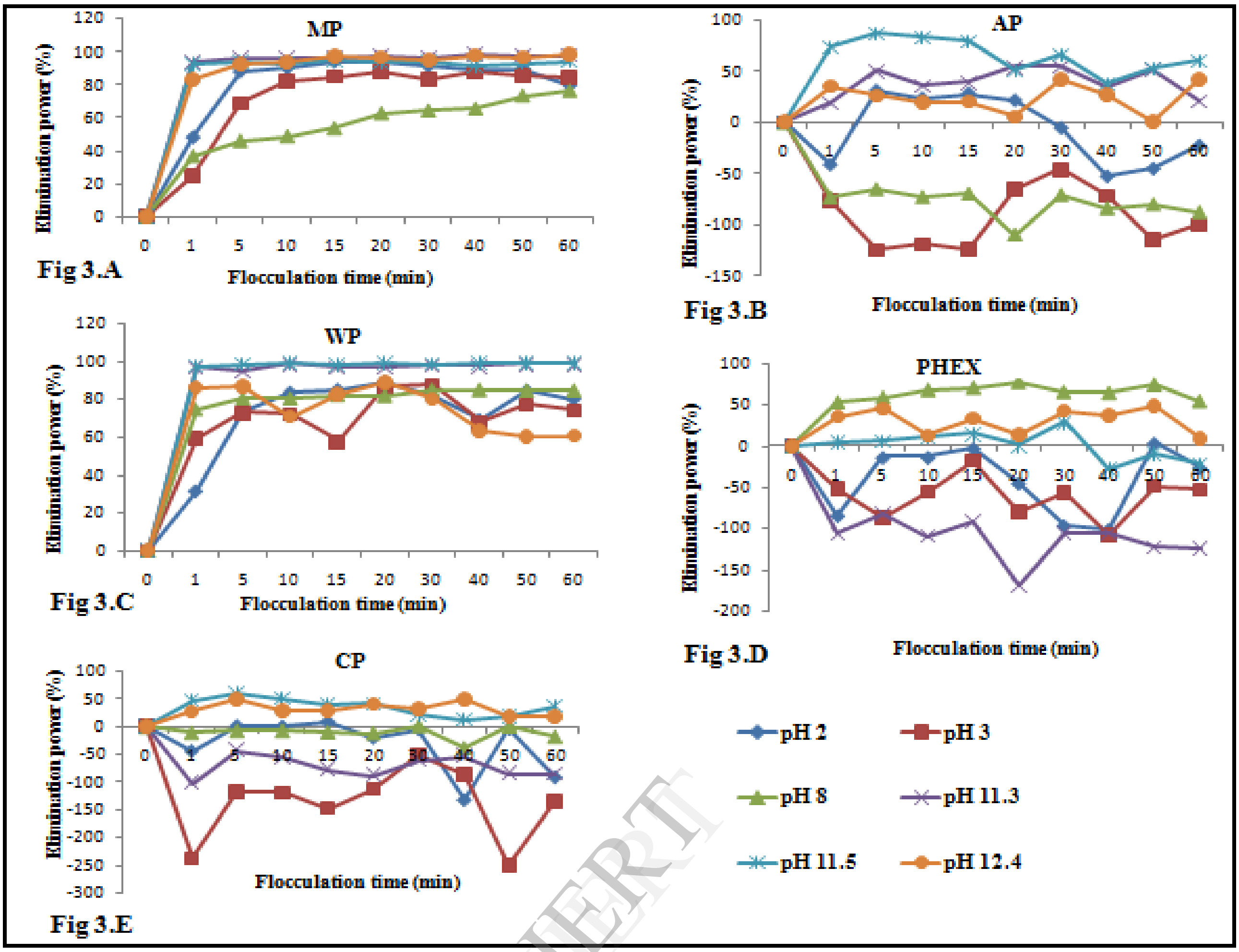

Fig. 3 . Effect of pH on the percent removal turbidity for a fixed dose of cactus extracts (2%) using standard jar test. In Fig 3.A, 3.B, 3.C, 3.D and 3.E we show the elimination power of turbidity by using MP, AP, WP, PHEX and CP flocculants, respectively.

B. Determination of the optimal flocculants dose

Once the optimal pH for each flocculant had been determined, experiments were performed varying the dose of coagulant between 0.5 and 5% in order to ascertain the influence on the coagulation-flocculation process and to determine the optimal dose. The optimum dose of a flocculant is defined as the value above which there is no significant increase in removal efficiency with further addition of flocculant. Fig. 4 plots turbidity elimination versus flocculants dose. The positive effect of using MP and WP could be clearly observed for the doses of 1% and 2% (Fig. 4A and 4B). However, for higher dose of MP and WP, no significant decrease of residual turbidity was observed. It has been suggested that optimum flocculation occurs when half the surface area of solid is covered with polyelectrolytes [28].At higher concentration the degree of flocculation decreases and the slow mixing time particles may be completely covered by the absorbed polymer layer. In the experiments with AP, CP and PHEX as coagulants aids, the lowest residual turbidity were 194, 360 and 441 NTU, respectively (Fig. 4C, 4D and 4E) from initial turbidities of 1000 NTU (High), for optimum conditions of pH and flocculants doses. From Figures 4C, 4D and 4E, it was seen that not a single residual turbidity satisfies the World Health Organization (WHO) limit of (5 NTU) for drinking water [29]. Excellent results were observed in case of MP and WP (Fig. 4A and 4B). Residual turbidities at 35 minutes were observed to be 2 NTU for the optimum dosage of 1% WP and 3 NTU for MP at 40 min of flocculation for an optimum dosage of 1% too. For MP and WP all the residual turbidities satisfies WHO's limit for drinking water at optimum conditions. It was clear from the present study that MP and WP showed a good flocculating activity in conjunction with lime for synthetic turbid water. The $Ca(OH)_2$ adsorbed on the surface of the particle and reduced the negative charge with its slightly positive charge. This new process has considerably reduced the chemicals dose of products usually used in coagulation flocculation treatment. Also sludge produced was less voluminous and readily biodegradable.

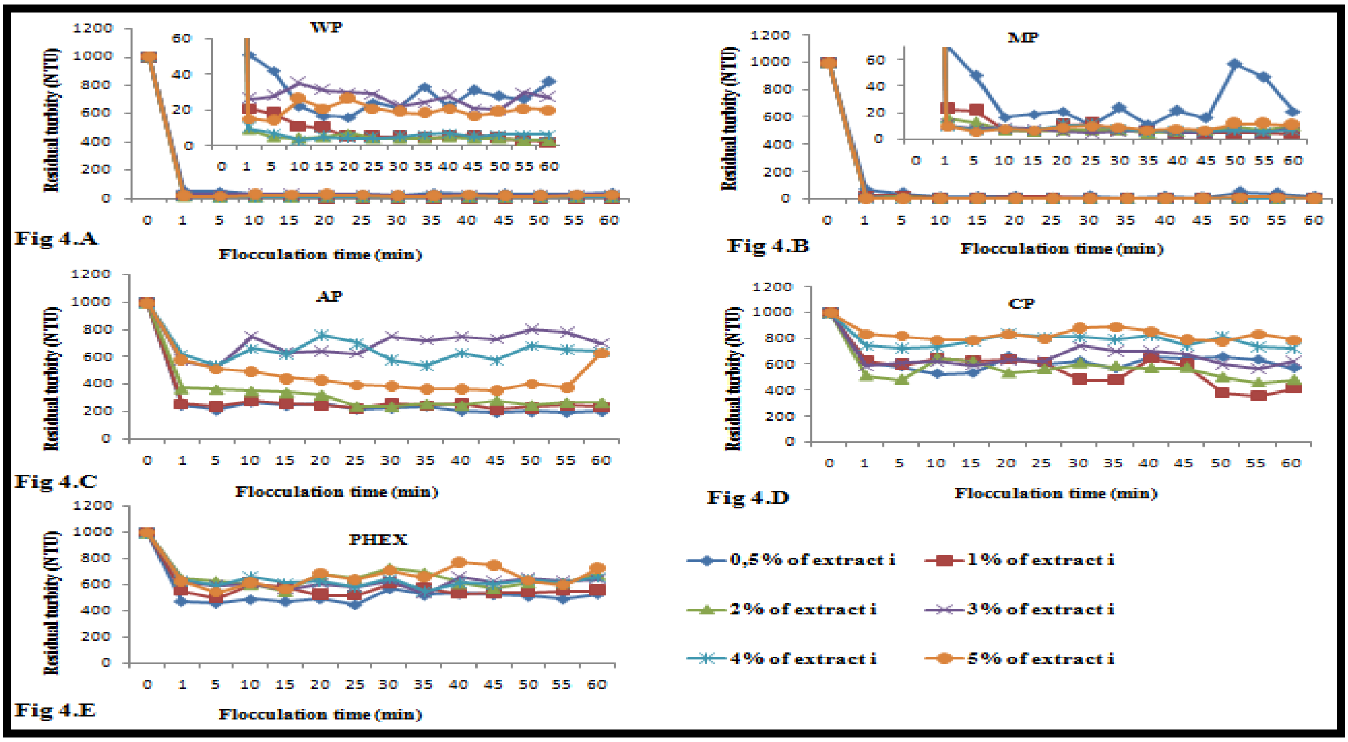

Fig. 4 . The effect of flocculants concentration on the final turbidity using doses of cactus extracts ranged from 0.5% to 5% (initial turbidity 1000 NTU) at optimum pHs. in Fig 4.A, 4.B, 4.C, 4.D and 4.E we show the residual turbidity using WP, MP, AP, CP and PHEX flocculants, respectively.

Conclusions

Based on the results obtained using MP and WP as flocculants and lime as coagulant, the following suggestions may be made for the physical–chemical treatment of a turbid water by coagulation- flocculation: Optimal dose of 1% of MP or WP at optimum pH ranged from 11 to 11.5. The turbidity removal efficiency for MP and WP were 98.5 and 99%, respectively. It is very clear from the literature and this work that pH and flocculant dose control of coagulation-flocculation are the most important parameters governing turbidity removal. The experimental results of present study confirm that the turbidity removal efficiency by these two bio-flocculants was excellent and satisfies WHO's limit. State and Central Government have created various rules and regulations against pollution. These regulations when fully effective will have profound effects, particularly on disposal practices of toxic industrial wastes. One obvious advantage of using renewable materials is the minimal net effect on global warming. Other advantages include biodegradability and sustainability.

Future studies will include additional tests and samples with varying mixing and times, flocculation, at various dosages for other potential coagulation/flocculation aids. In addition, sludge volumes will be estimated and economic costs estimated.

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