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Présenté par

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Thème

Optimisation de traitement d'une solution modèle par électro-flottation et ajout d'un coagulant : Le mucilage de la raquette d'*Opuntia ficus indica*

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Dedicate

I dedicate this work to

my Father "Youcef" and my Mother "Louiza"

my Bothers "Mouloud, Menad and Nadjib"

my Sisters "Zoulikha, Lahna and Sabiha"

my In-laws " MOHELLEBI" especially my Fiancé "Elyas"

my lovely nieces " Marwa and Ikram"

all my friends

Naima

List of abbreviations

ANOVA	:	Analysis Of Variance
$Al_2(SO_4)_3$	:	Aluminium sulfate
BBD	:	Box-Behnken Design
CO_2	:	Dioxide carbon
CAM	:	Crassulacean Acid Metabolism
cm	:	Centimeter
COD	:	Chemical oxygen demand
°C	:	Selcus degrees
DAF	:	Dissolution Air Flotation
DW	:	Dry Weight
EC	:	Electrocoagulation
EF	:	Electroflotation
FW	:	Fresh Weight
g	:	Gram
ha	:	Hectare
h	:	Hour
L	:	Liter
min	:	Minute
mg	:	Milligram
mL	:	Millilitre
mS	:	Millisemen’s
NTU	:	Nephelometric Turbidity Units
OFI	:	*Opuntia ficus indica*
O_2	:	Oxygen
pH	:	Hydrogen potential
RSM	:	Response Surface Methodology
TR	:	Turbidity Removal
UV	:	Ultra-Violet
v/v	:	Volume/ Volume
V	:	Voltage
%	:	Percentage

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Introduction

Environment and the natural equilibrium are the basis of social and human development. In this frame, and to insure a good strategy of a sustainable environment development, in its double aspect; natural resources control and different rejection, we want to gather every efforts for valorization of the natural resources (**Diaz and al., 1999; Zhang and al., 2006**).

Water concern is surely one of the main factors that are involved in the human development considering its influence on human lives. Among the new techniques for water and wastewater treatments, there is the use of natural coagulants, aiming at a better quality of treated water by reducing the use of chemicals (**Antov and al., 2012**). Namely, coagulation/flocculation step which is essential process in the treatment of both surface water and industrial wastewater, includes removal of dissolved organic species and turbidity from water most commonly via addition of conventional chemical based coagulants – alum, ferric chloride and synthetic organic polymers. While the effectiveness of these chemicals as coagulants is well-recognized (**Edzwald, 1993; Kang and al., 2003**) there are, nonetheless, disadvantages associated with their usage such as ineffectiveness in low-temperature water (**Haarhoff and Cleasby, 1988**), relatively high procurement costs, detrimental effects on human health, production of large sludge volumes.

Thus, in water treatment, the use of natural coagulants could be an option with many advantages over chemical agents, particularly the biodegradability, low toxicity and low residual sludge production (**Narasiah and al., 2002**). These advantages are especially augmented if the plant from which the coagulant is extracted is indigenous to local community. In recent numerous studies variety of plant materials has been reported as a source of natural coagulants (**Diaz and al., 1999; Miller and al., 2008; Raghuwanshi and al., 2002; Šćiban and al., 2009**).

The *Opuntia ficus indica* mucilage is a gummy substance produced in cells found both in chlorenchyma and parenchyma of the cladodes of *Opuntia* spp. that helps cactus to retain water (**Barbera and al., 1995**), it is an abundant and a low cost product. Tested as a coagulant for treating wastewaters, it was proved that it has an effect on decolorization, COD removal and turbidity abatement (**Adjeroud and al., 2015; Bouatay and Mhenni, 2014**).

Electrocoagulation and electroflotation (EC-EF) has been widely used in various wastewater. It has been applied for treatment of potable water **(Holt, Barton, Wark, & Mitchell, 2002**), urban, heavy metal, and colored waters **(Chen, Chen, & Yue, 2000; Jiang,**

Graham, André, Kelsall, & Brandon, 2002; Pouet & Grasmick, 1995). EC-EF assisted by *Opuntia ficus indica* pad juice, used as a naturel coagulant, enhanced the turbidity removal efficiency of the EC–EF technique by 15 %, thus, being more efficient than the EC-EF technique used solely(**Adjeroud, Dahmoune, Merzouk, Leclerc, & Madani, 2015**). However, there are no reports for the optimization of EC-EF parameters assisted by OFI mucilage using RSM.

This document will serve as an introduction to optimize the EC-EF water treatment method, by adding *Opuntia ficus indica* mucilage as a natural coagulant by response surface methodology (RSM) and to investigate the effect of some EC-EF operating parameters, such as: The various concentrations of OFI mucilage, pH, conductivity and voltage on turbidity removal efficiency.

For the elaboration of this work, the document is divided into two part: the first part includes generality on the bibliographical study and the second part contains the experimental procedures.

There is four chapters in the first part: chapter one presents general information on the prickly pear; its distribution, production and the interest of each part of this plant. Chapter two defines the mucilage, its reactional mechanism and its interest in the treatment of polluted waters. The third chapter relates to the definition of the EC.-EF technique and its principle advantages for the environment. Chapter four of this part defines RSM, its principal use and its interest for the treatment and the interpretation of the data.

Whereas the second part has two chapters. Materiel and methods as chapter one, includes first the plant harvest, followed by mucilage extraction and RSM optimization of EC-EF treatment assisted by OFI mucilage. The second chapter focuses on the interpretation of the different obtained results.

Chapter I

1. Origin and geographical distribution.

Opuntia ficus indica (OFI) is a cactus originary of the arid and semi-arid areas of the United States and Mexico, its introduced in North Africa at the 16th century by Spanish (**Pottier-Alapetite, 1979**).

In North Africa (Morocco, Algeria and Tunisia), since several decades, the OFI made an integral part of the coastal landscape and insular areas, as the facility testifies some with which its adapted to the climatic conditions (**Mulas and Mulas, 2004a**). Today, the OFI proliferate on arid and perish-desert grounds of the five continents **(Arba and al., 2000)**.

OFI is a succulent plant of type CAM (Crassulacean Acid Metabolism) (**Nobel, 2002**), it presents morphological and physiological adaptations, they letting to resist to the dryness, hot heats and winds violent (**Mulas and Mulas, 2004; Sudzuki, 1995**).

2. Botanical classification.

Cataceae are calceiform angiosperms dicotyledonous dialypetalous in the order of caryophyllaceous (**Wallace and Gibson, 2002**). They are classified among the Xerophytes plants because they can persist very long periods of dryness's; Succulent because their faculties to store water in vegetative fabrics which take a spongy aspect. The cactaceae family includes approximately 300 species divided into four sub-genera (**Arba, 2000**)

Wallace and Gibson (**2002**) reported following classification:

Kingdom	Plantae
Under-kingdom	Spermaphyte
Division	Angiosperme
Class	Dicotylédone
Sub-class	Caryophyllale
Family	Cactaceae
Sub-family	opuntiadieae
Sub-genus Kind	Opuntia Opuntia
Species	*Opuntia ficus indica*

3. Description of OFI.

These hardy perennials arborescent succulent, with height of 3 to 5 m, thick and woody trunk, the length of stems is in average 30 to 60 cm or more. Its flattened in the shape of cladodes with a matt green color; these articles have a length of 15 to 30cm and a width of 30 to 60 cm, they are covered small areolas and spines. The flowers with their yellow color, are marginal on the top of the cladodes and they are hermaphrodites. The flowers color becomes reddish with the approach of the senescence of the plant. Its fruits are fleshy bais and ovoid or pyriform equipped with spines. The fruits are generally greenish to yellowish in maturity. The pulp is always juicy with yellow-orange color, red or crimson, strewn with many small seeds (**Habibi,2004**).

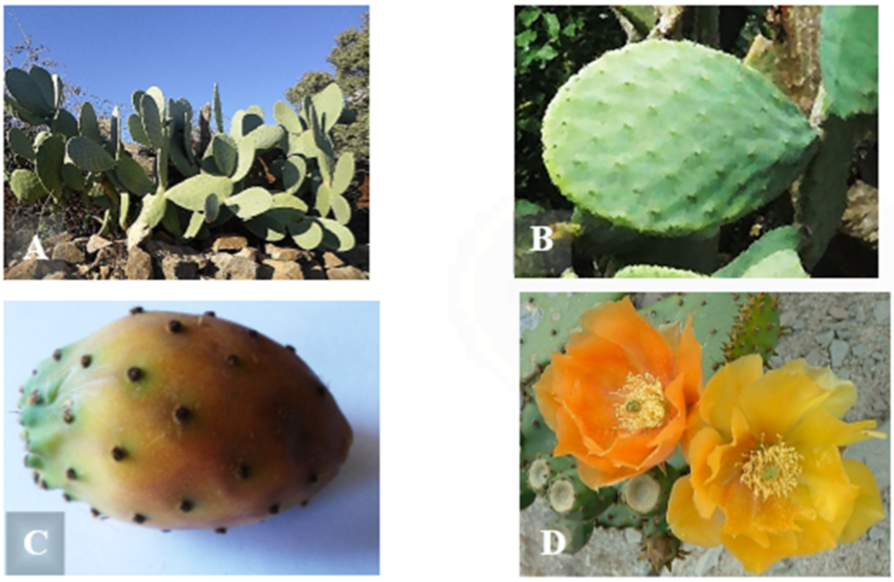

Figure (01): *Opuntia ficus indica*, (A) the plant, (B) the cladodes, (C) fruit, (D) the flower (**Habibi, 2004**)

4. Species and varieties of the OFI

The fruits of OFI, which are the object especially trade local, are present on the market in July until the end of September. There are many varieties of OFI, which are distingued in two great groups:

- **Spineless cactus :** are often domesticated and cultivated on limited surfaces.
- **Thorny cactus:**are most widespread because they resist the destruction by the cattle.

There are also several intermediate varieties between the spiny form and inerm. This variability is due to the influence of various conditions pattern soil and climatic of the cactus culture areas **(LOUDYI, 1994**).

Arba and al. (**2000**) have makes it possible to distinguish certain varieties by their morphological characters from where:

- The shape of fruit (ovoid, round, lengthened, elliptic);
- The color of flower (yellow, orange or red);
- The color of pulp (green, yellow, orange or red);
- Periods of flowering and maturity (late or early);
- Organoleptic characteristics of the fruits (sweetened, juicy, consistency…).

5. Metabolism of the plant

OFI is qualified by xerophyte plant, juicy, generally thorny, with fabric mucilaginous and aqueous, the stomata are protected to decrease surface perspiration and the roots to absorb the dew of the night (**Brulfert and al., 1987**). OFI is CAM (Crassulacean Acid Metabolism) plant because it has the capacity to fix the carbon dioxide (CO_2), and to release oxygen (O_2) during the night and close its stomata during the day (ordinary reverse of the mesophyte plants which photosynthesis during the day). In the succulent plants, at night the stomata open and allow the fixation of the dioxide carbon (CO_2) in chloroplast fabrics. The metabolism of the carbohydrates via glycolysis produces oxaloacetate, which is transformed into malate. This last will be stored in the vacuole, during the day, it breaks up into pyruvate and releases the carbon dioxide (CO_2) and water (H_2O) on the level of the chlorophyllian fabrics which serve for the continuation of photosynthesis according to the cycle of Calvin (**Lüttge, 1993; Sutton and al., 1981**) (Fig. 02). This mechanism allows a less water loss per evapotranspiration in the hottest periods.

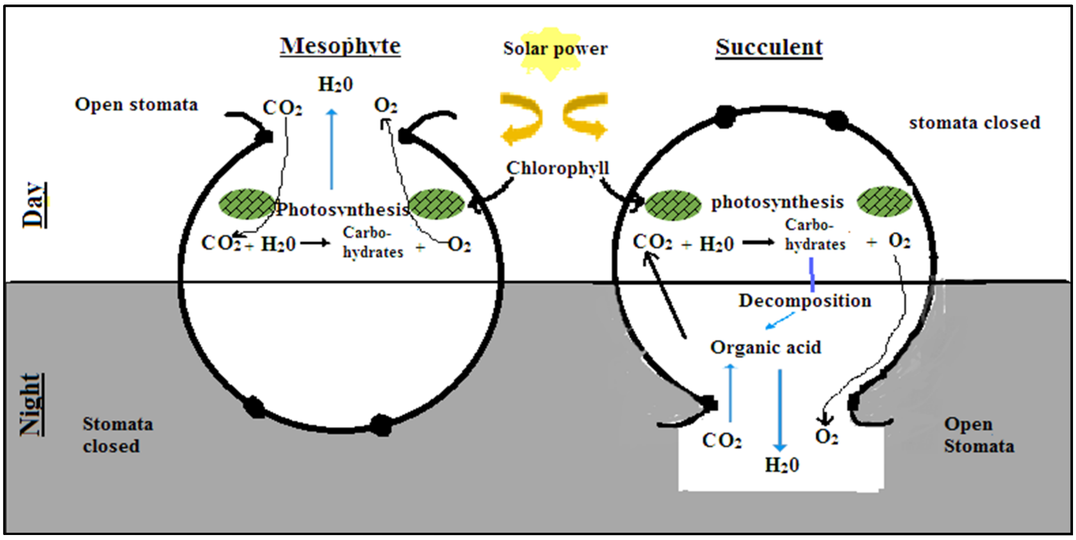

Figure (02): Photosynthetic cycle of the CAM plants type (**Habibi, 2004**).

6. Interests and uses of the OFI.

OFI has a potentially interesting, it is primarily cultivated for the production of its fruits (**Nobel and al., 1992**). The ensuing tale show the principal uses of the different parts of the OFI.

Table I. The principals uses of the different parts of the OFI.

Part	Uses	References
Fruits	- Consumed in a fresh state; - Conditioned industrially and stabilized by various methods (cold, drying, heat); - The peels of the fruits can replace the vitamin E used in margarine conservation as an antioxidant.	**Arba, 2009; Hamdi, 1997; Kunyanga et al., 2009 ; Mohamed-Yasseen et al., 1996 ; Chougui et al., 2015**
Flowers	- Used principally in medicinal remade (Controlling diuretic, Dysfunction of the prostate and his action on hypoglycemic and hypolipidemic)	**(Palevitch et Craker, 1994; Cassano-Piche et al., 2009**)
Cladodes	- Consumed by different strata of people(USA, Mexico); - Wastewater treatment; - Against the soil erosion; - Yogurt fermentation; - Production of the red dye color “carmin” ; - Source of bioenergy and biogas; - Additive in the manufacture of shampooing, astringent, cream…	**(Bouatya et Mhenni, 2014 ; Càrdenas et al., 1997 ; Miller et al., 2008 ; Sàenz et al., 1992 ; Abidi et al., 2009 ; Ben Salem et Smith, 2008 ;E.Pimienta et al., 1993 ; Barbera et al ;, 1995 ; E.Pimienta, 1994 ; Cantwell, 1991 ; Russell et Felker, 1987)**

7. Production

In certain countries OFI is currently cultivated in an intensive way, it is the subject of a culture with whole share, for the fodder exploitation(**Sudzuki-Hillis, 1995**). Indeed, it is a plant, which all the parts can be consumed. However, the production of fruits remains the aspect more developed (Table. II).

Statistical studies carried out by **Inglese and al**. (**2002**), show that Mexico is the principal producer country of prickly pear, with a production rate of more than 345 000 tons/year.
In Europe, Italy is the country more invested in the culture of OFI. Generally used as fruit and fodder. Silica has the first place with more than 90% of the total production.

In the Mediterranean, and especially in the Maghreb, Morocco is the large producer with a surface of occupation by the prickly pear of more than 46 000 ha. The production is estimated at 57 000 tons with an average output of 1,2 ton/ha. The output in Tunisia is estimated from 250 to 300 quintals of fruits/ha (**Loudyi, 1994**), whereas in Algeria, the production is artisanal and major the part of the quantity of product is self-consumption or marketed in an anarchistic way.

Table II. Principal producer countries and uses of the OFI in the world (**Nobel, 2003**).

Country	Surface (ha)	Collect (tons/years)	Use principal
Argentina	900	8 000	Fruit
Bolivia	1 300	3 500	Fruit, dye
Chile	1 200	9 000	Fruit
Israel	400	7 000	Fruit
Italy	7 500	80 000	Fruit
Mexico	70 000	400 000	Forage, Fruit, dye
North Africa (Morocco, Tunisia, Algeria)	20 000	/	Forage, Fruit
South Africa	200- 1500	1 500	Fruit
The United States (California)	200	3 600	Fruit

One finds also plantations commercial of OFI in Argentina, in Brazil, in Chile, in Algeria and in South Africa In certain areas, inside and outside the surface of natural distribution, one uses stems of OFI. This plant is currently integrated in the strategies of fight against the turning into a desert in various countries (**Nefzaoui and al., 2012; Sudzuki, 1995**).

8. Ecological factors limiting the culture and the production of the cactus.

The OFI present adaptations physiological and morphological of which the juicy stem (water stores during the season of the dryness) and sheets (the presence of the spines makes it possible to avoid the water losses per evaporation), thus allowing this plant to resist the difficult conditions of the arid and semi-arid areas.

The prickly pear has a great adaptation to the most hostile conditions **(Loudyi, 1995; Nobel, 2002**).

- Dry and hot climate, even arid, it tolerates the low temperatures being of -5°C to -10°C.
- Light, sandy-muddy grounds low in organic matter 0,1°C to 1,8%, having a pH slightly acid 5,1 to 6,7.

Chapter II

1. Cladode

The cladode term is starting formed from the old Greek: Clad « Klados », who mean "branch" and Ode "eïdos", which means "form, aspect".

The cladodes are specialized branches represent adaptations of the further shaft, having the appearance of a sheet and providing the same functions (photosynthesis and respiration, reserves). The cladodes are short and consist of a single internode (**Boullard, 1997**). The cladodes contain a viscous liquid and they are covered with a cuticle that has the ability to reflect light and minimize evaporation, these forms allow the plant to develop a heat resistance

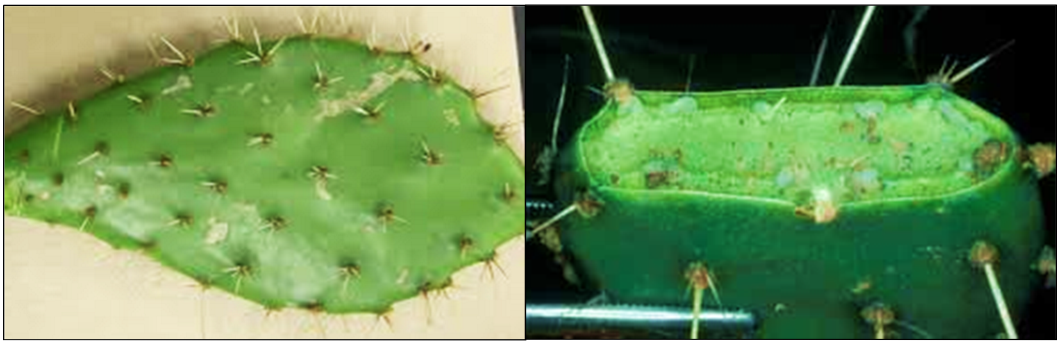

Figure (03): Photography of OFI cladode outside and the inside part.

1.1. Cladode morphology

The study carried out by **Malainine and al**.(**2001**) with using a microscope transmission balyage have shown that the cladodes consists essentially of epidermal cells, parenchymal and chlorenchyma as shown in (fig.3).

- **Epidermis**: is a coating of the primary cloth cell. It is provided with impermeable thin cuticle in lipid nature interrupted by openings called stomata. The outer surface is composed by areolas, which contain multiple glochides (**Habibi, 2004**).
- **Chlorenchyma**: is the continuous outer cortex of several layers of cells arranged perpendicularly beneath the epidermis of the cladode (**Wallace and Gibson, 2002)**.
- **Parenchyma:** is the most abundant cellular cloth of the cladode, composed of cellulose microfibrils associated strap between them and are interlaced together (**Malainine and al., 2001**).

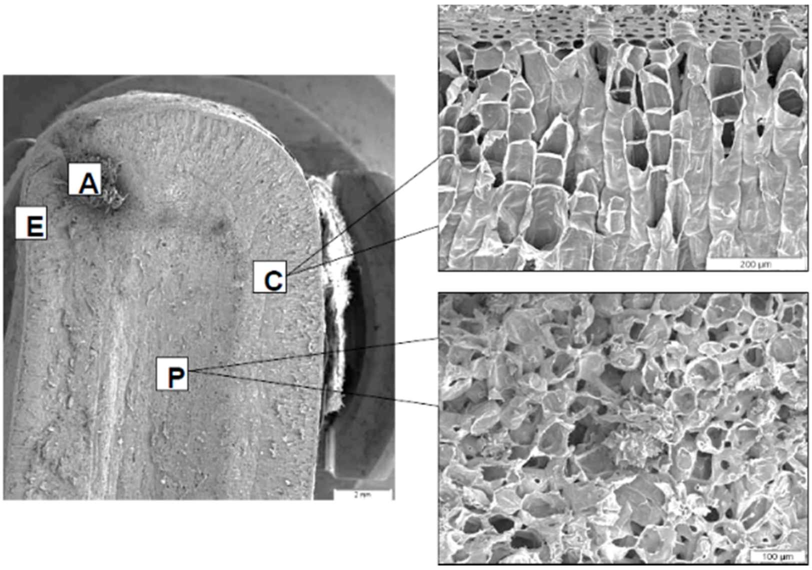

Figure (04) : SEM Micrographs obtained by electronic scan microscopy of a transverse section of a cladode of OFI (**Malainine and al., 2003**).
E: epidermiques cells, **A**: areolas, **P**: cells of parenchyma, **C**: cells of chlorenchyme.

1.2. Chemical Composition

The cladodes of OFI are mainly rich in water, cladodes are considered a high energy source because of their high content of total sugar by comparing it with other fruits. The various chemical constituents of a highly increased significantly during their growth stages (**Hadj Sadok and al., 2009**), The main chemical component contents are summarized in the table (III).

Table. III: Major cladodes Compounds of OFI (**Càrdenas and al., 1997**; **Hadj Sadok and al., 2009**).

Constituent	Contents
Water content (% FW)	93
Minerals (% DW)	13.84
Total fiber (% DW)	42.99
Total sugar (%DW)	5.79
Sugars reducing (%DW)	1.95
Total nitrogen matter (%DW)	2.51
Titrable acidity (%)	0.33

2. Mucilage

Mucilage is a gummy substance produced in cells found both in chlorenchyma and parenchyma of the cladodes of *Opunti*a spp that halps cactus to retain water (**Barbera and al., 1995**).

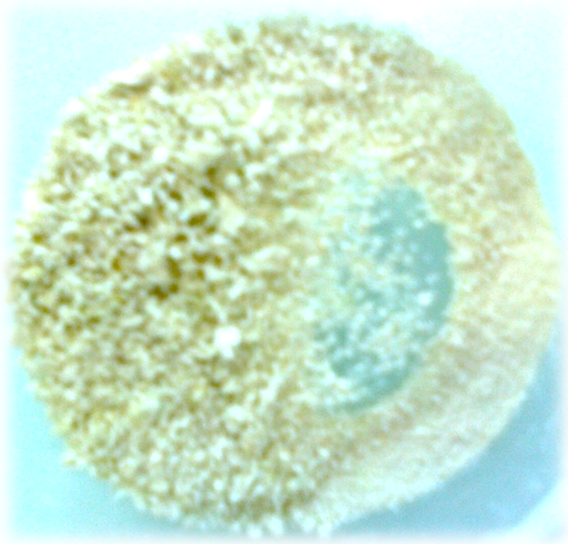

Figure (05): *Opuntia ficus indica* mucilage (Photo of Laboratory 3BS)

Mucilages are complex polymeric substances of carbohydrate nature, with a highly branched structure (**Medina- Torres and al., 2003)**, which contains varying proportions of L-arabinose, D-galactose, L-rhamnose and D-xylose, as well as galacturonic acid in different proportions. It composed approximately of 55 sugar residues without uronic acid, (**Amin and al., 1970**). The proportion of the different OFI mucilage compound are summarized in the table (IV).

Tableau IV. The principals tenor of the mucilage compounds (**Nobel and al., 1992; Trachtenberg and al, 1981**).

Sugar	**Tenor (%)**
Arabinose	67.25
Galactose	6.27
Rhamnose	5.43
Xylose	20.41
Uronic acid	19.5

2.1. Chemical structure

Trachtenberg and Mayer (1981) is the first suggested structure of the mucilage, he describes the molecule as a linear repeating core chain of (1-4) linked a-D-galacturonic acid and (1-2) linked **β**-l-rhamnose with lateral chains of (1-6)- **β**-D-galactose attached to O-4 of rhamnose residues. **McGarvie and Parolis**, (**1981a, b**) suggest that the galactose residues present substituents in the positions O-3, or double substitution in O-3 and O-4 (Fig. 04) and that the composition of the outlying chains is complex; at least 20 different types of oligosaccharides. **Majdoub and al.** (**2001**) reported that in OFI the water-soluble polysaccharide fraction with thickening properties represents less than 10% of the water-soluble material.

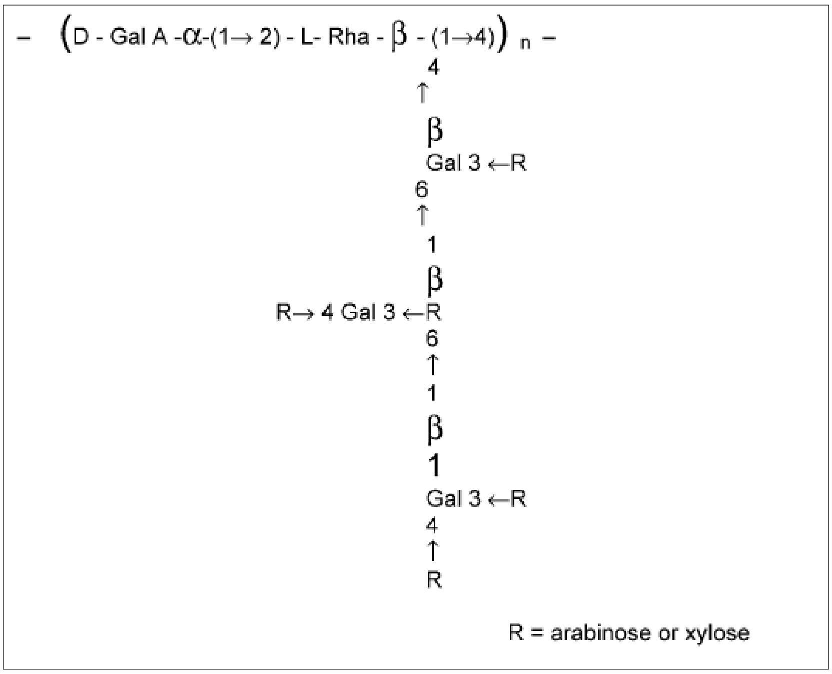

Figure (06): Proposed partial structure for the OFI mucilage (**McGarvie and Parols, 1981**).

2.2. Application and uses of mucilage

The mucilage of OFI, which contain a biopolymer with interesting characteristics from the rheological point of view, but also because if its coagulating- flocculating capabilities (**Rivera- Corona and al., 2014**).

Càrdenas and al. (**1998**) has pointed out that, for centuries, nopal juice has been added as an organic adhesive to lime in order to restore and protect historical buildings in Mexico. **Adjeroud and al**. (**2015**) reported that the treatment of effluent in a concentration 0.0016ml/L OFI juice pads assisted by EC-EF could remove 87% of turbidity. **Sadok and al**. (**2014**) have showed that the OFI juice pads can stimulate the lactic bacteria, in the fermentation stage.

Another property of nopal mucilage is its use in foods; the use as a fat replacer in foods is mentioned and also as a flavor binder (**Càrdenas and al., 1997)**.

Rwashda cited by **Garti** (**1999**) studied the emulsifying capability of the OFI gum. It found that this gum:

- Reduced surface and interfacial tensions,
- Stabilized oil–water emulsions,
- Formed small oil droplets,
- Absorbed onto oil–water interfaces and does not contribute to the viscosity of the systems,

Garti and Leser (**2001**) reported that OFI gum exhibited strong emulsifying capabilities in dilute oil–water emulsions.

Chapter III

1. Definition

1.1. Electroflotation:

It is a simple process that floats pollutants to the surface of the water body by tiny bubbles of hydrogen and oxygen gases which generated from water electrolysis (**Bhaskar and al., 1984; Chen, 2004)**.

1.2. Electrocoagulation

Electrocoagulation is the process of destabilizing suspended, emulsified, or dissolved contaminants in an aqueous medium by introducing an electric current into the medium. In its simplest form, an electrocoagulation reactor that may be made up of an electrolytic cell with one anode and one cathode (**Emamjomeh and Sivakumar, 2009**). The electrocoagulation (EC) has recently been suggested as an alternative to conventional coagulation (**Emamjomeh and Sivakumar, 2009**).

1.3. EC-EF

Singh and al. (**1998**) developed the electrocoagulation / Electroflotation process to remove color and pollutants from wastewater. This process involves applying an electric current to sacrifice electrodes inside a reactor reservoir where the current generates a coagulating agent and gas bubbles. The ions coagulate with pollutants in the water, similar to the addition of coagulating chemicals such as chloride aluminum and chloride ferric, and allow for easier removal of the pollutants by sedimentation and flotation. In a general sight, EC-EF is a technology based on the concepts of electrochemical cells, specifically known as ‘‘electrolytic cells. In an electrolytic process a source of direct current is connected to a pair of electrodes immersed in a liquid that serves as the electrolyte (**Emamjomeh and Sivakumar, 2009**).

2. Principe of electrocoagulation- electroflotation

The process of EC-EF was realized with using Aluminum or iron electrode materials which are usually used as anodes and cathode, the dissolution of which produces hydroxides, oxyhydroxides and polymeric hydroxides. These are usually more effective coagulants than those used in chemical dosing: they are able to destabilize colloidal suspensions and emulsions, to adsorb, neutralize or precipitate dissolved polluting species, and finally to form flocs that can be removed by either settling /filtration or flotation. In EF, settling is the most common option, while flotation may be achieved by H_2 or assisted by air injection (**Holt and al., 2002**).

The metal ions can react with the OH^- ions produced at the cathode during gaseous H_2 evolution, to yield insoluble hydroxides, which adsorb pollutants out of the solution. It also contributes to coagulation by neutralizing the negatively charged colloidal particles which have been reported to be more compact than sludge obtained by chemical methods (**Merzouk and al., 2010**). The electrocoagulation process is as follows:

- At the anode,

$M \rightarrow M^{n}+ + né$…………………………………………………………………………(1)

- At the cathode,

$n\ H2O + ne \rightarrow \frac{n}{2} H_2 + n\ OH^-$……………………………………………………..…………...(2)

where, M=anode material and n=number of electrons involved in the oxidation/reduction reaction.

Ions of soluble metals as A1 and Fe are generated at the anode and react with the hydroxide ions formed at the cathode, and the metal hydroxides are produced as expressed by reaction. (3).

$M^{n+} + nOH^- \longrightarrow M(OH)_n$………………………………………………………………….(3)

These insoluble metal hydroxides react with the suspended and/or colloid solids and precipitate.

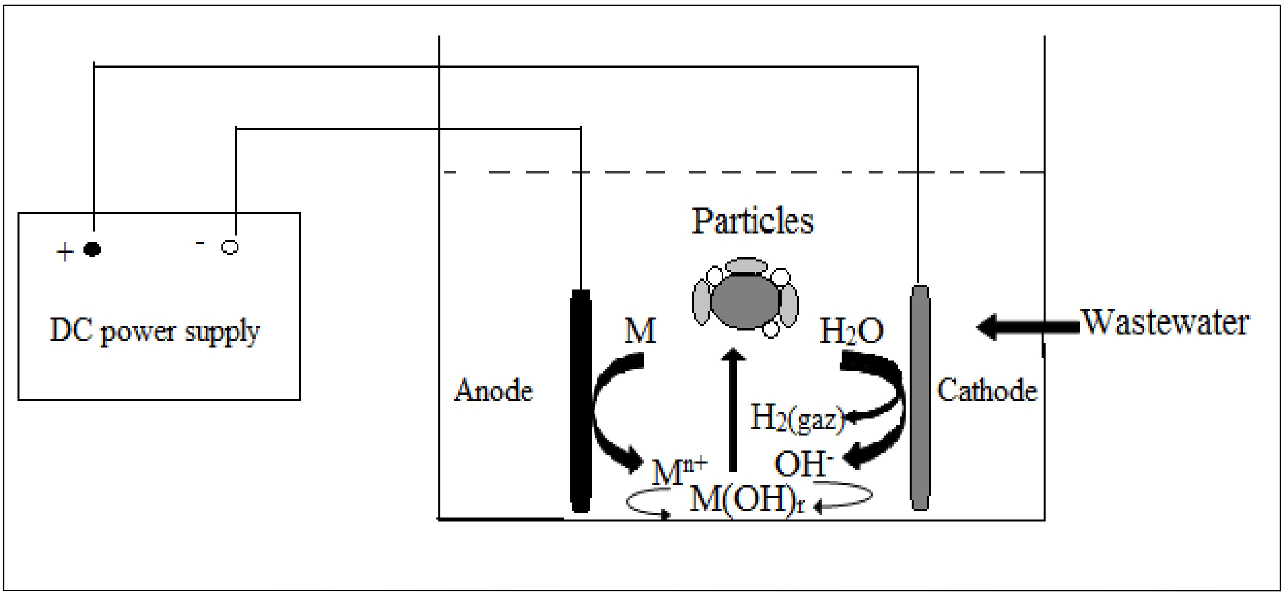

Figure (07): Diagram of the principle of electroflotation-electrocoagulation (**Merzouk, 2003**).

3. Factors affects the EC-EF

3.1. Electrodes Arrangement

The electrode can be arranged in horizontally or vertically.

3.1.1. Horizontally

The electrodes can be arranged in different patterns. Usually a plate electrode is installed at the bottom, while a screen electrode is fixed at 10–50 mm above the plate electrode as shown in Fig.8. (**Chen and Chen, 2010**).

This arrangement of the electrode leads to the reduction on the effect of the treatment with EF, because one of the electrode which is the upper screen electrode will be faces the water flow, while the bottom electrode does not interact with the flow directly. Therefore, the bubbles generated at the bottom electrode cannot be dispersed immediately into the water being treated. This not only decreases the availability of the effective small bubbles, but also increases the possibility of breaking the flocs formed previously, affecting the flotation efficiency. In addition, if the conductivity is low, energy consumption will be unacceptably high due to the large interelectrode spacing required for preventing the short circuit between the upper flexible screen electrode and the bottom plate electrode. This renders EF economically unfavorable in competing with the conventional DAF. Moreover, the maintenance of the electrodes system is a problem because the screen electrode is easy to be twined by undesirable deposits such as fabric, and difficult to clean once twined (**Chen and Chen, 2010**).

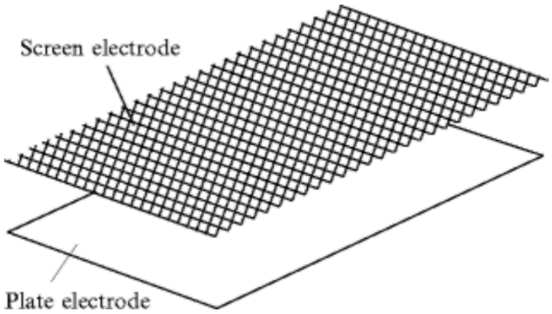

Figure (08): Horizontal electrodes arrangement(**Chen and Chen, 2010**).

3.1.2. Vertically

The electrodes can also be arranged vertically as shown in Fig.9. (**Chen and Chen, 2010**). For such an arrangement, both the anodes and the cathodes are designed as plate shape, and therefore they can be fixed easily. However, the bubbles generated trend to rise along the surfaces of the electrodes, which is often observed in the electrocoagulation process . Therefore, the bubbles generated at both electrodes can be dispersed into water rapidly and attach onto the flocs effectively, ensuring high flotation efficiency. **Chen and al.** (**2000**) resulting in a quick bubbles coalesce and thereby affecting the flotation efficiency.

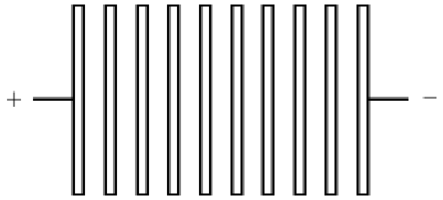

Figure (09) : Vertical electrodes arrangement (**Chen and Chen, 2010**).

3.2. Type electrode materials

3.2.1. Cathodes

Metals and alloys are usually selected as cathodes for hydrogen evolution in EF. For most cases, stainless steel is a good choice because it is cheap and readily available. Nickel is known to have low over potential for hydrogen evolution. Therefore, usage of a nickel cathode can save energy consumption. Titanium is expensive, but it is very stable. Therefore, this metal can be selected as a cathode material in treatment of corrosive wastewaters (**Chen and Chen, 2010**). Cathodes undergo a scaling by carbonate deposits, which reduce their effectiveness gradually.

3.2.2. Anodes

Compared with cathodes, available anodes are much less; because severe electrochemical corrosion may occur when common, metals and alloys are used as anodes. Actually, the lack of stable and cheap anodes was one of the key factors that limited the wide application of EF in industry. (**Chen and Chen, 2010**)

Graphite, PbO2, and Pt are among the most common no consumable anode materials.Graphite and PbO2 anodes are cheap and easily available, and thus have been widely investigated for oxygen evolution in EF (**Burns and al., 1997; Ho and Chan, 1986; Hosny, 1996**). However, the durability of graphite and is poor. Its service life is only 6-24 months in part due to the oxidation. In fact, the stability of PbO2 also poor (**Ho and Chan, 1986**). 68

mg/L after 24 h of electrolysis. Therefore, graphite and PbO2 are not good oxygen evolution anodes for EF.A few researchers reported the use of Pt as anodes for EF (**Ketkar and al;, 1991; Poon, 1997**). They are much more stable than graphite and PbO2. However, their known high costs make large-scale industrial applications impracticable.

Usually, aluminum or iron plates are used as electrodes in the electrocoagulation process (**Chen and al., 2002**). Aluminum has as favors compared to iron not to color the treated solution (**Abuzaid and al., 2002; Öğütveren and al., 1994; Wilcock and al., 1996**).

3.3. Current density

Is the important parameter influencing the performance and economy of the efficacy of electrocoagulation process (**Kobya and al., 2006**). The gas bubbles depends also on the current density (**Ketkar and al., 1988; Ketkar and al., 1991)**. A decrease of gas bubble sizes was found with the increase of current intensity.

3.4. pH

It has been established that the influent pH is an important operating factor influencing the performance of the EC and EF processes (**Chen, 2004)**.

The hydrogen bubbles are smallest at neutral pH. For oxygen bubbles, their size increase with pH. It should be noted, however, the cathode materials affect the size of the hydrogen bubbles, and so do the anode materials. The bubble sizes obey a log-normal distribution (**Fukui and Yuu, 1985)**.

Llerena and al. (**1996**) found that the decrease or increase of pH results the increase or decrease of hydrogen bubbles. The size of electrolytic bubbles has been found to be pH dependent. In alkali medium, bubbles of oxygen are bigger than those of hydrogen. Under acidic conditions, otherwise holds.

3.5. Salinity

In order to decrease the resistivity of the effluent to treat and thus decrease the power consumption for the same production of agent of coagulation, it can be desirable to add a standard electrolyte NaCl (**Öğütveren and al., 1994; Wilcock and al., 1996**). However, the improvement of the results depending little on the added quantity for good quality of the treatment (**Abuzaid and al., 2002**). It should be noted that this addition must be more significant if one wishes to carry out in more one disinfection per *in situ* hypochlorite formation (**Tsai and al., 2002**).**Advantages and disadvantage**

4.1. Advantages

The benefits of EF include

- They are simple equipment and easy operation (**Merzouk and al., 2010**);
- Short reactive (electrolyte) period (**Merzouk and al., 2010**);
- Flotation of the pollutant to the solution surface where it can be more easily collected and removed (**Merzouk and al., 2010).** EF, as an electrochemical version of flotation, usually has higher separation efficiency than the conventional DAF due to its much smaller hydrogen and oxygen bubbles generated electrolytically**;**
- Simplicity, efficiency, environmental compatibility, safety, selectivity, flexibility and cost effectiveness (**Bayramoglu and al., 2007; Mohammad and al., 2004**);
- The reduction of sludge generation (**Alinsafi and al., 2005**);
- The minimization of the addition of chemicals and little space requirements due to shorter residence time (**Belkacem and al., 2008**);
- The cost-effective of the EC-EF as chemical dosing (**Bayramoglu and al., 2007; Holt and al., 2002**);
- Technique which practically applicable to solve environmental pollutions problems (**Merzouk and al.,2010**);

4.2. Disadvantages

Belkacem and al. (2008) say that the main deficiency is the lack of dominant reactor design and modeling procedures and the energy consumption that is very important. **Mollah and al.** (**2001**)**,** the drawbacks of EC-F are:

- The ‘sacrificial electrodes’ are dissolved into wastewater streams as a result of oxidation, and need to be regularly replaced;
- The use of electricity may be expensive in many places;
- An impermeable oxide film may be formed on the cathode leading to loss of efficiency of the EC unit;
- High conductivity of the wastewater suspension is required;
- Gelatinous hydroxide may tend to solubilize in some cases.

Chapter IV

It is important to improve the performance of the systems and to increase the yield of the processes without increasing the cost. The method used for this purpose is called optimization (**Baş and Boyacı, 2007**).

1. Theory and definition

Response surface methodology was developed by Box and collaborators in the 50s (**Marcos and al., 2006**)..

RSM has important application in the design, development and formulation of new products, as well as in the improvement of existing product design. It defines the effect of the independent variables, alone or in combination, on the processes. In addition to analyzing the effects of the independent variables, this experimental methodology generates a mathematical model called response surface (RSM) (**Zangeneh and al., 2002**).

RSM includes three techniques or methods (**Montgomery and Myers, 1995**):

- Statistical experimental design, in particular, two-level factorial or fractional factorial design;
- Regression modelling techniques; and
- Optimization methods.

RSM can be viewed from three major standpoints (**Cornell, 1990**):

- If the system response is rather well-discovered, RSM techniques are used to find the best (optimum) value of the response;
- If discovering the best value is beyond the available resources of the experiment, then RSM techniques are used to at least gain a better understanding of the overall response system;
- If obtaining the system response necessitates a very complicated analysis that requires hours of run-time and advanced computational resources then a simplified equivalent response surface may be obtained by a few numbers of runs to replace the complicated analysis.

2. Definition of some terms

Before beginning the discussion on the applications of response surface in the optimization of analytical methods, it is pertinent to introduce and define some key terms.

- **Experimental design**

Experimental design is a specific set of experiments defined by a matrix composed by the different level combinations of the variables studied (Bezerra, Santelli, Oliveira, Villar, & Escaleira, 2008).

- **Coded factor levels**

Experimental designs are often written in terms of coded variables (**Hibbert, 2012**). In screening designs, the factors are usually examined at two levels (–1, +1) (**Vera and al., 2014**). We can choose two, three or several levels according to needs for the study (**Goupy and Creighton, 2006**).

- **Experimental domain**

It is defined by the minimum and maximum limits of the experimental variables studied (**Bezerra and al., 2008**).

- **Responses or dependent variables**

Responses or dependent variables are the measured values of the results from experiments. Residual is the difference between the calculated and experimental result for a determinate set of conditions. A good mathematical model fitted to experimental data must present low residuals values (**Bezerra and al., 2008**).

- **Response surface designs**

With the experimental results of a response surface design, a polynomial model, describing the relation between a response and the considered factors, is build. Usually a second-order polynomial model is constructed.

Afterwards, the model can be interpreted graphically and/or statistically. Graphically, the model is visualized by drawing 2D contour plots or 3D response surface plots. A 2D contour plot (Fig.10.a) shows the isoresponse lines as a function of the levels of two factors, while a 3D response surface plot (Fig.10.b) represents the response in a third dimension. From such plots, often the best or optimal conditions are derived. However, one should be aware that, in case three or more factors are considered, a plot as in Fig.10.

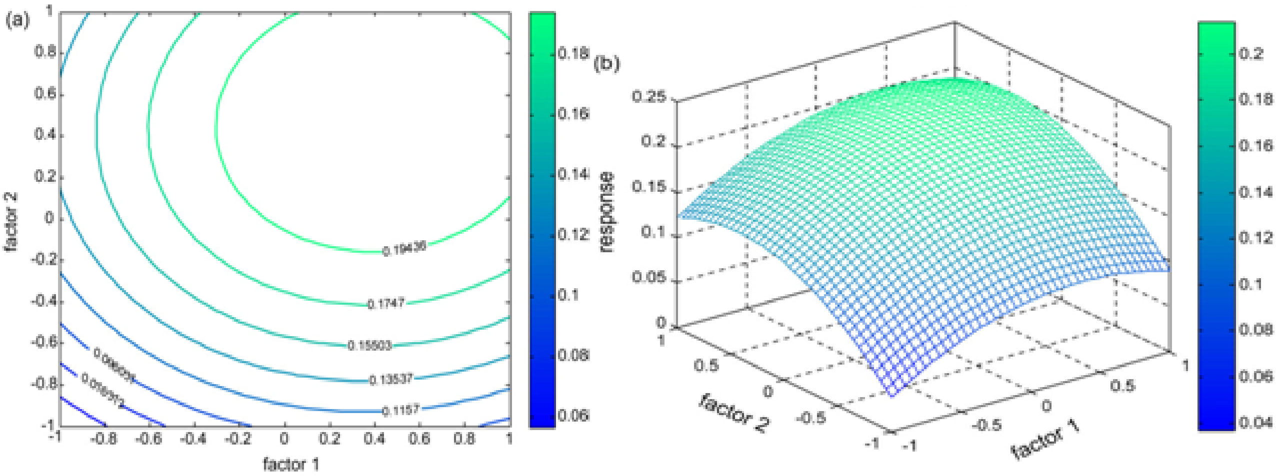

Figure (10): (a) 2D contour plot, and (b),3D response surface plot.

- ### Central composite design

A central composite design (CCD) combines a two-level factorial design with a star design and center points. The star and factorial points can lie equidistant from the center (circumscribed design), or the star points can lie within the space of the factorial design (inscribed design) or they can lie on the faces of the factorial design points (faced). This point is selected to obtain several properties, such as ratability or orthogonality, in order to fit the quadratic polynomials. The CCD is a better alternative to the full factorial, three-level design as it demands a smaller number of experiments while providing comparable results **(Hibbert, 2012**).

3. Steps for RSM application

Some stages in the application of RSM as an optimization technique are as follows:

3.1. Screening of variables

Numerous variables may affect the response of the system studied, and it is practically impossible to identify and control the small contributions from each one. Therefore, it is necessary to select those variables with major effects. Screening designs should be carried out to determine which of the several experimental variables and their interactions present more significant effects (**Bezerra and al., 2008; Dejaegher and Vander, 2011; Hibbert, 2012; Vera and al., 2014**).

3.2. Choice of the experimental design

The simplest model which can be used in RSM is based on a linear function. For its application, it is necessary that the responses obtained be well fitted to the following equation:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \mathcal{E} \tag{4}$$

where k is the number of variables, β_0 is the constant term, β_i represents the coefficients of the linear parameters, X_i represents the variables, and ε is the residual associated to the experiments.

Therefore, the responses should not present any curvature. To evaluate curvature, a second-order model must be used. Two-level factorial designs are used in the estimation of first-order effects, but they fail when additional effects, such as second-order effects, are significant. So, a central point in two-level factorial designs can be used for evaluating curvature. The next level of the polynomial model should contain additional terms, which describe the interaction between the different experimental variables. This way, amodel for a second-order interaction presents the following terms:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{1\leqslant i\leqslant j}^{k} \beta_{ij}\ X_i X_j + \mathcal{E} \tag{5}$$

where βij represents the coefficients of the interaction parameters.

In order to determine a critical point (maximum, minimum, or saddle), it is necessary for the polynomial function to contain quadratic terms according to the equation presented below:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X^2\ + \sum_{1\leqslant i\leqslant j}^{k} \beta_{ij}\ X_i X_j + \mathcal{E} \tag{6}$$

where βii represents the coefficients of the quadratic parameter.

To estimate the parameters in Eq. (6), the experimental design has to assure that all studied variables are carried out at in at least three factor levels. Thus, two modeling, symmetrical response surface designs are available. Among the more known second order symmetrical designs are the three-level factorial design, Box–Behnken design (BBD), central composite design, and Doehlert design. These symmetrical designs differ from one another with respect to their selection of experimental points, number of levels for variables, and number of runs and blocks (**Bezerra and al., 2008**).

3.3. Evaluation of the fitted model

The mathematical model found after fitting the function to the data can sometimes not satisfactorily describe the experimental domain studied. The more reliable way to evaluate the quality of the model fitted is by the application of analysis of variance (ANOVA). The central idea of ANOVA is to compare the variation due to the treatment (change in the combination of variable levels) with the variation due to random errors inherent to the measurements of the generated responses. From this comparison, it is possible to evaluate the significance of the regression used to foresee responses considering the sources of experimental variance (**Bezerra and al., 2008**).

Lack of fit test is another way to evaluate the model. It expresses the variation of the data around the fitted model. A model will be well fitted to the experimental data if it presents a significant regression and a non-significant lack of fit. In other words, the major part of variation observation must be described by the equation of regression, and the remainder of the variation will certainly be due to the residuals. Most variation related to residuals is due to pure error (random fluctuation of measurements) and not to the lack of fit, which is directly related to the model quality (**Bezerra and al., 2008**).

3.4. Determination of the optimal

The surfaces generated by linear models can be used to indicate the direction in which the original design must be displaced in order to attain the optimal conditions. However, if the experimental region cannot be displaced due to physical or instrumental reasons, the research must find the best operational condition inside the studied experimental condition by visual inspection. *Residual* is the difference between the calculated and experimental result for a determinate set of conditions. A good mathematical model fitted to experimental data must present low residuals values (**Bezerra and al., 2008**)..

4. Advantages and disadvantages of RSM

RSM has several advantages compared to the classical experimental or optimization methods in which one variable at a time technique is used (**Bezerra and al., 2008**).

- Firstly, RSM offers a large amount of information from a small number of experiments. Indeed, classical methods are time consuming and a large number of experiments are needed to explain the behavior of a system.
- Secondly, in RSM it is possible to observe the interaction effect of the independent parameters on the response.

Chapter V

1. Chemical reagents and equipment

All chemicals and the reagents used in the experiments are collected in Annex I.

2. Vegetable materials

2.1. Collect

Nopal pads (Cladodes) were collected in January 2015 in the region of Aokas, area (300m altitude) which is located on the Mediterranean littoral, at the east of Bejaia city. The plant variety without spines was selected following its abundance in the area. The sample was composed of 02 cladodes.

Figure (11): OFI cladode photographs (a) before, (b) after pretreatment.

2.2. Pretreatment

The Nopal pads were washed with tap water, then with distilled water to remove all impurities (dust, glochids,..), then dried at room temperature during 30 min. Their physical characteristics were then determined including dimensions (wide, length, and diameter…). The Table.VII shows the physical characteristics of the studied cladodes.

Table.V. The principal characteristics of the cladodes used for the treatment

Parameters	**Characteristic**
Form	Ovoid
Weight(g)	899 ± 163.78
Thickness(cm)	0.82 ± 0.2
Wide (cm)	19.35 ± 1.51
Long (cm)	36.45± 2.08
areoles numbers	144 ± 2.5

3. Chemical analysis

3.1. Determination of moister (AOAC, 1980).

The moisture content was determined by drying cladodes (2 g) at 105°C until a constant weight was reached. Then the moisture content was calculated from the following equation:

Moisture = 1- (Dry Weight/Fresh Weight).

3.2. pH

The pH was directly given using a pH-meter (pH211.HANNA Instruments, Romania) on the crushed of cladode.

4. Extraction of mucilage

4.1. Grinding

After washing, the cladodes were crushed in a blender after dissection. Then, the mash of cladodes was homogenized with distilled water at 1:3 solid to liquid ratio at ambient temperature and agitation for 4 hours. The mixture afterwards was filtered through a fine cloth (**Sepùlveda and al., 2007 modified**).

4.2. Extraction steps (Sepùlveda and al., 2007 modified)

The filtarate obtained after filtration was precipited in Ethanol 95% (v/v) in the proportion of 1:3 liquid to liquid, after precipitation the mucilage was separated, the solvent used was then evaporated at 40°C in oven. The recovered mucilage was frozen and lyophilized (Telstar, Spain) under a pression of 488 Barr and at temperature of (-50°C). The obtained mucilage was conserved at 4°C. The protocol is summarized in figure (12):

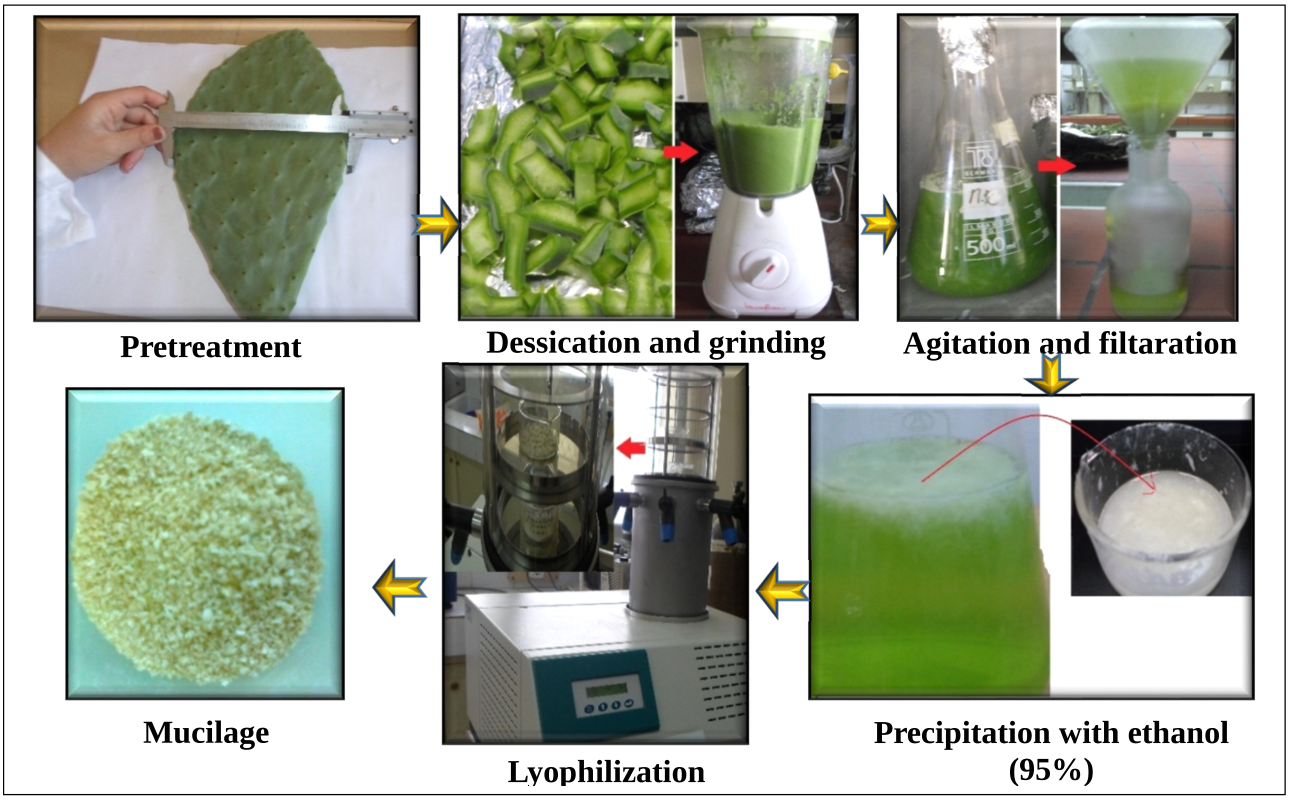

Figure (12): Mucilage extraction stages (photography taken at the 3BS Laboratory).

5. Preparation of turbid water model and its treatment with EC- EF by addition of OFI mucilage.

5.1. Preparation the synthetic wastewater

The synthetic wastewater was prepared using 2000 mL of tap water to cover the aluminum electrodes in the EC–EF unit, to a concentration of 300 mg/L of silica gel (Woelm Pharma) to simulate highly turbid industrial discharges (**Adjeroud and al., 2015; Merzouk and al., 2009**).

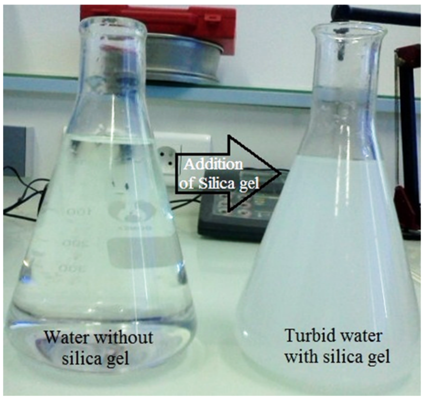

Figure (13): The appearance of the water before and after addition of silica gel (photography taken at the 3BS Laboratory).

5.2. Preparation of the mucilage solution.

After weighing the necessary quantity of mucilage for the manipulation, the mucilage powder was re-dissolved in 18ml of distilled water with adding 2ml of NaOH (1M). The solution was stirred at room temperature until solubilisation was complete (**Kaewmanee and al., 2014 Modified**).

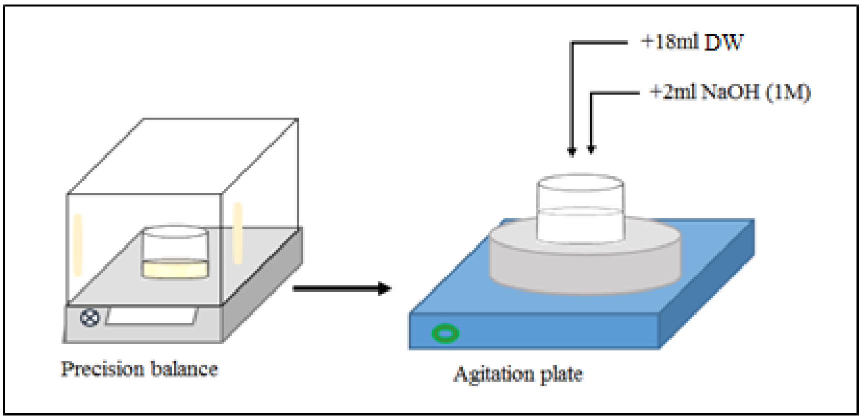

Figure (14): Mucilage weighing and dissolution.

5.3. Preparation of the EC-EF batch unit

The experimental equipment is schematically shown in Fig.15. The electrocoagulation-electroflotation (EC-EF) unit consisted of an 4 L electrochemical reactor with two aluminum electrodes of rectangular shape (27 mmX17 mmX1mm), corresponding to S = 4.59 cm^2 electrode surface area, installed vertically in the middle of the reactor. The electrodes were connected to a DC power supply (Statron Typ 3217, Germany) providing 0–30 V (0–10 A). The distance between electrodes was 1 cm. All the runs were performed at room temperature. In each run 2000 mL of the synthetic wastewater was decanted into the electrolytic cell and samples were taken at 4.5 cm above the bottom of the reactor and measured over time after sedimentation. Neither centrifuging nor filtration was performed. All experiments were repeated three times.

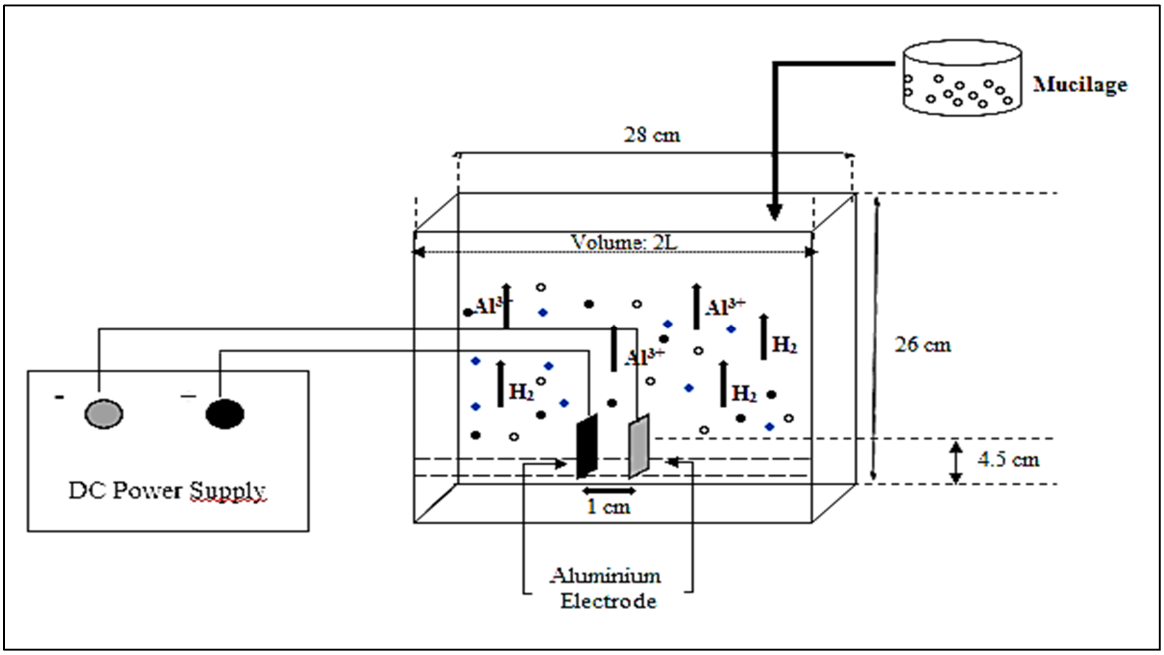

Figure (15): EC-EF Batch unit assisted by OFI mucilage

6. Analysis

Water samples of water to be treated were recovered before the treatment, they were analyzed and they were considered as controls. After application of the treatment, the sample recovery was made at regular time; each 5 min, then the samples were analyzed. The analyses carried out were turbidity, pH and conductivity.

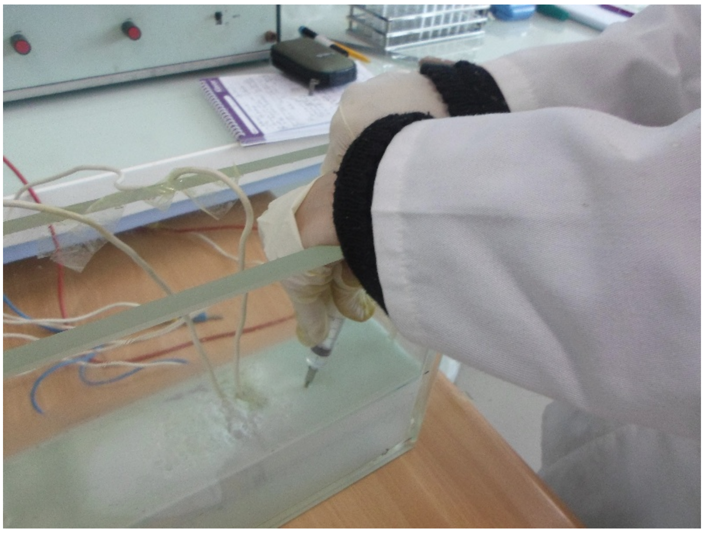

Figure (16): Samples recovery prior to turbidity analysis (photography taken at the 3BS Laboratory).

6.1. Turbidity measurement

Turbidity, which is caused by fine suspend particles of clay, silt, organic and inorganic matter and microscopic organisms, is an important indicator to evaluate the safe use of water (**Howard and al., 2001**). Thus, turbidity reduction can vastly improve the effectiveness of disinfection method.

Samples turbidity was determined by UV–VIS spectrophotometer (Spectro Scan 50, Japan). Silica gel concentration was estimated from its absorbance characteristics at maximum wavelength (λ_{max}) of 740 nm (**Adjeroud and al., 2015**). The calculation of turbidity removal efficiency (TR) after electrochemical mucilage free treatment or mucilage assisted treatment was performed using the following formula:

$$\boldsymbol{TR}\ (\%) = \frac{\mathbf{C_0} - \mathbf{C}}{\mathbf{C_0}}$$

Where, C_0 and C are concentrations of silica gel before and after EC–EF process in mg/L respectively.

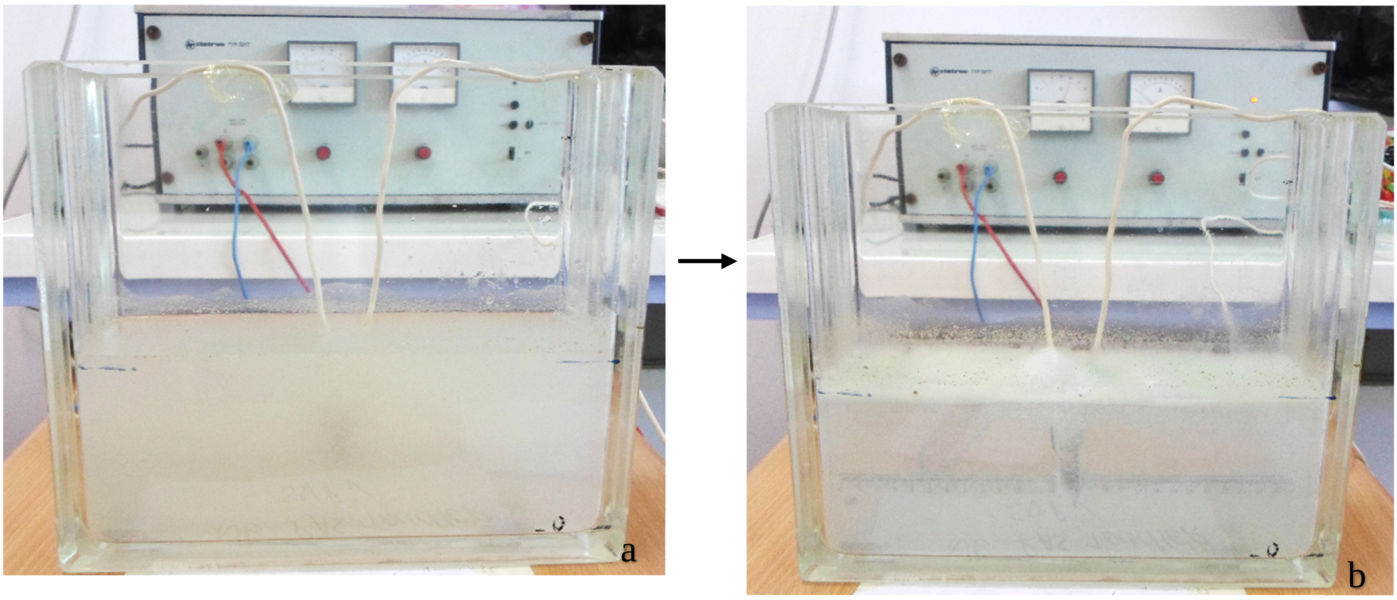

Figure (17): Wastewater in EC-EF reactor of the treatment by EC-EF (photography taken at the 3BS Laboratory).

(a) before, (b) after (at the right treatment)

6.2. pH Measurement

The effect of some parameters as pH is very important to investigated the EC-EF time (**de Oliveira da Mota and al., 2015**).

The pH was measured with a pH-meter (pH211.HANNA Instruments, Italy). The desired pH was attained by adding HCl (1M) or NaOH (1M) (**Miller and al., 2008**). The interval of pH chosed was limited from 5 to 10.

6.3. Conductivity Measurement

The conductivity was measuring with multi-parameter (Extech instruments , French) where indicate the desired conductivity was attained by adding NaCl (1M) (**Adjeroud and al., 2015**). The conductivity effect was investigated between the values of 1.8mS/cm and 3.4 mS/cm.

7. Experimental design

7.1. Preliminary study

The objective of the preliminary study is to determine the appropriate parameter intervals required to optimized the EC-EF performance by the response surface methodology (RSM). According to the study already made (**Adjeroud and al., 2015; Chen and al., 2000; Merzouk and al., 2009**) the most parameter influencing on the EC-EF efficiency are: pH, voltage and the conductivity.

For optimization of OFI mucilage assisted by EC-EF treatment, the influences of the process parameters were, firstly separately investigated in single-factor experiments to limit the total experimental work (Fig.18). When one variable was not studied, it was kept constant. For the enhancement of the EC-EF treatment assisted by OFI mucilage the constants values for the determination of the mucilage concentration effect are for voltage, conductivity and pH 20V, 1.31 mS/cm and 7.67 respectively. To investigate the effect of voltage the OFI mucilage concentration was kept constant at 2.5 mg/L, pH and conductivity were equal to 7.67 and 1.31mS/cm respectively. To inspect the effect of pH, the concentration of the mucilage, voltage and conductivity were fixed at 2.5 mg/l, 20V and 1.31 mS/cm, respectively. To examine the effect of conductivity, the concentration of the mucilage, voltage and pH were fixed at 2.5 mg/l, 20V and 9 respectively. To investigate the effect of the voltage, concentration of the mucilage, conductivity and pH were fixed at 2.5 mg/L, 2.5 mS/cm and 9, respectively. During all the trials, the constant values for the inter-electrode distance and initial synthetic wastewater concentration were 1 cm and 300 mg/L, respectively (**Adjeroud and al., 2015; Belkacem and al., 2008**).

A response surface methodology (RSM) based on a Box–Behnken Design (BBD) for turbidity removal efficiencies (TR) was conducted to optimize the process (**Ahmad and Langrish, 2012**).

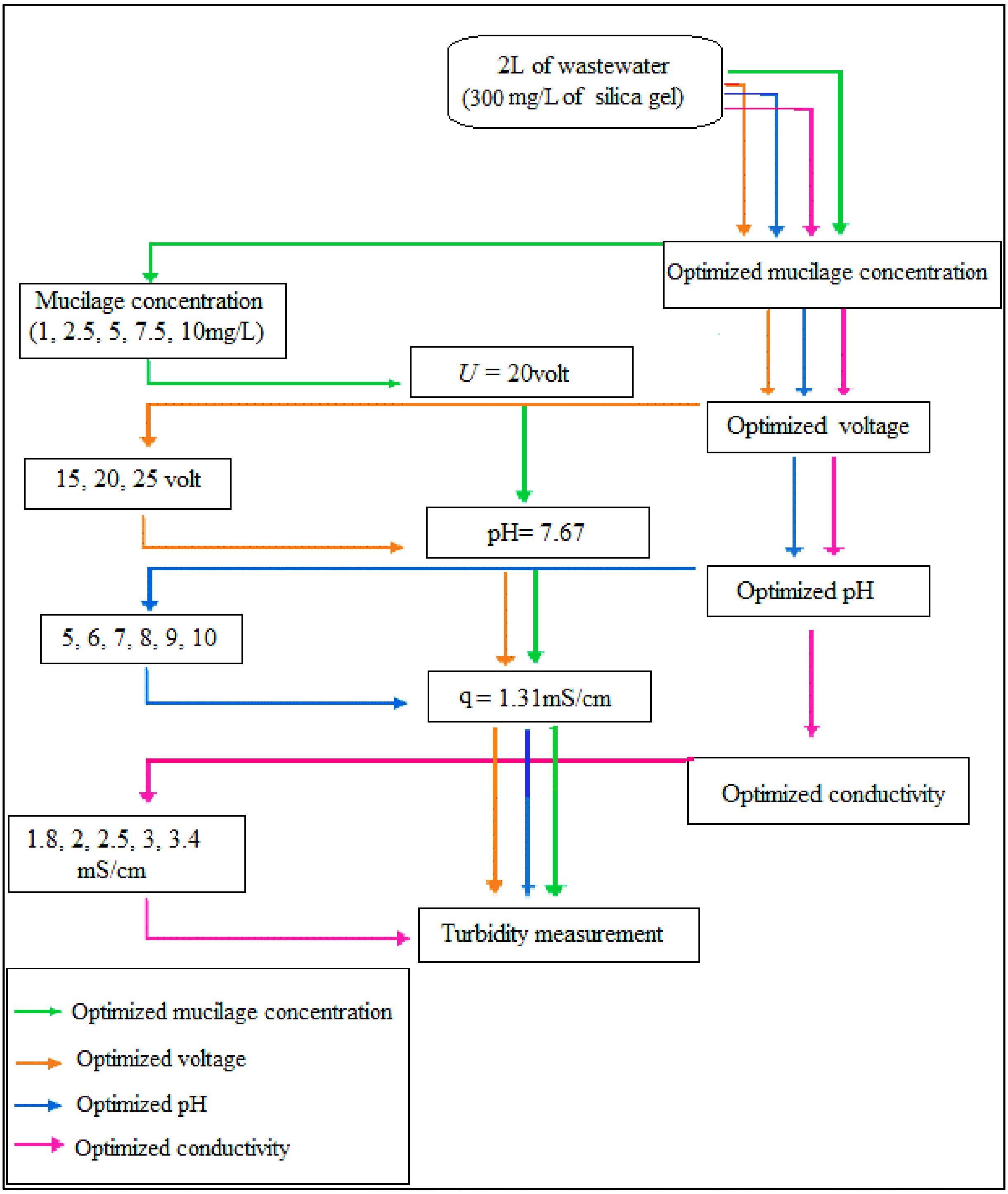

Figure (18): Single-factor experimental procedure (Preliminary study).

7.2. RSM optimization

On the basis of the preliminary trials and to determine the preliminary range of the treatment, the factor levels corresponding to each independent variable X_1 (Mucilage concentration), X_2 (pH), X_3 (voltage) and X_4 (conductivity) were chosen. The turbidity removal (TR) was the dependent variable response. Then, a response surface methodology based on a Box–Behnken Design (Minitab, version 9.0.4.1, USA) for Nopal pad mucilage assisted EC–EF treatment was conducted to optimize the process.

To investigate the effect on the turbidity removal efficiency, three levels effects by a second order BBD with 27 points, and 3 replications of the center points were studied. Factor levels were coded as -1 (low), 0 (central point or middle) and +1 (high), respectively.

Table. VI. The independent variables and their levels influencing the turbidity removal efficiency.

Factors	**Variables**	**Coded levels**		
		-1	**0**	**+1**
X_1	Mucilage (mg/L)	1	3	5
X_2	pH	7	8.5	10
X_3	Voltage (V)	15	20	25
X_4	Conductivity (mS/cm)	2	2.5	3

8. Statistical analysis

Data obtained from the BBD was statistically analyzed using ANOVA for the response variable in order to test the model significance and suitability, $p < 0.05$ and $p < 0.01$ were taken as significant and highly significant levels, respectively. Box–Behnken Design (Minitab, version 9.0.4.1, USA) software was used to construct the BBD model and to analyze all the results.

Chapter VI

Generally, the treatment by electrocoagulation-electroflotation (EC-EF) on the turbidity removal efficiency is influenced by several parameters such as the pH, conductivity, voltage and the coagulant concentration when added.

Aluminum is the most frequently used material for electrodes in the electrocoagulation process (**Chen and al., 2002**) because it is cheap and readily available. EC–EF produces coagulants in situ by electrical dissolution of either aluminum or iron ions. The metal ions generation occurs at the anode by oxidation reaction, at an appropriate pH they can form wide ranges of coagulated species and metal hydroxides that destabilize and aggregate the suspended particles or precipitate and adsorb dissolved contaminants. Hydrogen gas, that would help to float the flocculated particles, is released from the cathode where reduction reaction occurs (**Chen, 2004; Chen and al., 2000; Kobya and al., 2006).** In the case of aluminum, main reactions are as follows (reactions 5 and 7):

- Anode : $Al_{(s)} \rightarrow Al^{3+}_{(aq)} + 3\ e^{-}$ (5)
- Cathode : $3H_2O_{(1)} + 3\ e^{-} \rightarrow \frac{3}{2} H_{2(g)} + OH^{-}_{(aq)}$ (6)
- Hydrolysis reaction: $Al^{3+} + 3H_2O_{(1)} \rightarrow Al(OH)_{3(s)} + 3H^{+}_{(aq)}$ (7)

The entire experimental work is divided into two large parts. The first part consists of mucilage extraction that will be used as natural coagulant to improve the electrocoagulation-electroflotation treatment. The second part was initiated by preliminary experiments that helped determined lower, middle and senior design variables used in the response surface methodology (RSM). This first stage of the preliminary experiment is to find the appropriate intervals corresponding to each parameter, in order to be used for RSM oprimization. RSM had shown to be a powerful tool for the optimization of experimental conditions to maximize the various responses. To check the suitability (validation) models, a second part consisted of a treatment according to optimal conditions predicted with RSM mathematical models. The obtained experimental data were compared with the values predicted by the regression model. Effectiveness of the addition of the mucilage of *Opuntia ficus indica* as a coagulant in the treatment with EC-EF was compared with the conventional method without mucilage extract addition (control).

1. Chemical analysis

1.1. Test of Humidity of the OFI cladodes

The humidity rate of the OFI cladodes was 93.68% ± 0.35. This value is close to that found by **Hadj Sadok and al.** (**2009**) and **Valente and al**. (**2010**) that were 93% and 95%, respectively. The difference can be explained probably by the variations of the climatic conditions as well as the stage of maturation or the defects of handling.

1.2. pH

The pH of the studied cladodes was 4.42. This result was similar to that find by **Rodriguez-Felix and Cantwell.** (**1988**) and **Hadj Sadok and al.** (**2009**) which are 4.6 and 4.7, respectively. This difference can be explained of soil type and climatic conditions the defects of handling.

2. Preliminary study

2.1. Single factors analysis method

Table (IX) shows the results of the single-factor experiments carried out for preliminary optimization of the EC-EF technique and the turbidity removal efficiency values. The influence of each factors was studied. The presumed factors influencing the effectiveness of the EC- EF treatment are shown in Table (IX). The factors selected influences effectively the turbidity removal efficiency**.**

Table. VII. Results of single-factor experiments for OFI mucilage assisted EC–EF water treatment.

Mucilage concentration (mg/L)	**TR (%)**	**pH**	**TR (%)**
0	58.11 ± 9.96	5	39.07 ± 1.42
1	72.24 ± 4.98	6	50.65 ± 6.83
2.5	88.86 ± 7.62	7	78.99 ± 0.3
5	76.06 ± 2.88	8	80.68 ± 4.37
7.5	67.82 ± 10.84	9	88.39 ± 2.24
10	47.47 ± 9.63	10	64.11 ± 1.49

Voltage (V)	**TR (%)**	**Conductivity (mS/cm)**	**TR (%)**
15	80.61 ± 9.321	1.8	90.36 ± 2.08
20	89.97 ± 4.48	2	87.78 ± 1.55
25	81.18 ± 1.92	2.5	93.93 ± 1.02
		3	87.87 ± 3.68
		3.4	85.98 ± 7.01

2.1.1. Influence of the voltage on the turbidity removal efficiency

Several studies suggest that the electric voltage is one of the most important variable in EC-EF treatment. It is strongly dependent on the current density, the conductivity of the water to be treated and the inter-electrode distance and the surface state of electrodes (**Chen and al., 2002**). In fact, **Mollah and al.** (**2001**) reported that the current density is the most important parameter because it affects bubble size and their formation rate. The high current density increases bubble formation rate and decreases their size **(Lakshmi and Sivashanmugam, 2013).**

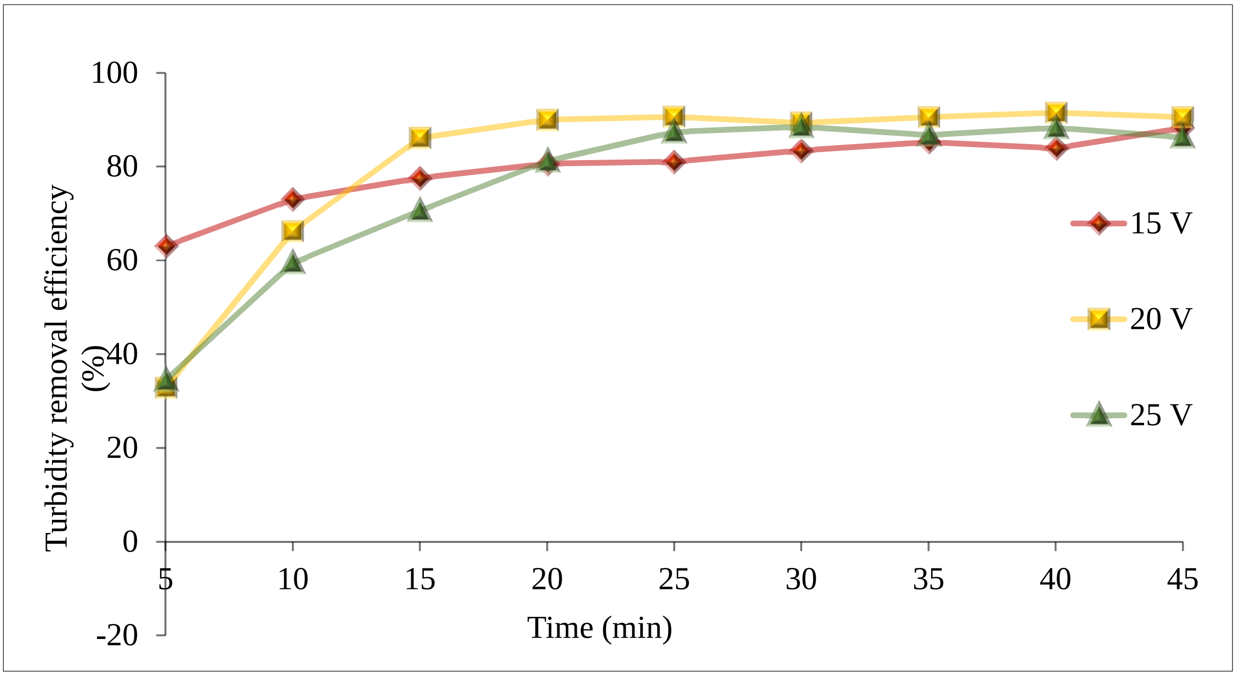

Figure (19): Effect of applied voltage *U* on turbidity removal efficiency: initial silica gel concentration C_0 = 300 mg/L, initial pH = 7.67, inter-electrode distance d = 1 cm, conductivity k = 1.31 mS/cm.

The figure above showed the effect of voltage on turbidity removal efficiency. To investigate the effect of voltage on turbidity removal efficiency, different values of voltage were tested (15, 20 and 25 V). EC-EF was carried out under preselected experimental conditions: initial pH 7.67, distance between electrodes 1 cm, conductivity 1.31 mS/cm and initial synthetic wastewater concentration 300 mg/L. According to the figure, we note that turbidity removal reached 89.97 % at 20 V which is equivalent to 74.62 ± 6.92 mg/L, this means that initial turbidity was, reduce by 4 fold. Whereas at 15 and 25 V the initial turbidity was diminished by 3 and 2 fold where the concentration was 117.69 ± 2.90 and 148 ± 1.13 mg/L, respectively. Then the maximal value of turbidity removal was recorded at 20 V and at run time of 20 min.

This performance was similar to that found by **Belkacem and al**. (**2008**) the turbidity removal found was 70.8 % at 20 V comparing with the different voltages tested (10, 15, 20 and 25 V). **Cho and al.** (**2010**) has shown that the electric voltages has an effect on the NH_4-N removal and that the best result is when the voltage 7 V was applied, the yield of the elimination was 99%.

Thus, at different voltage values, we observed:

- At 15V, the production of turbidity removal was weak; this result can be explain by the great size of formed bubbles because the weak anodic dissolution of Al and H_2 gas production.
- At 20V, the anodic dissolution of Al increased, resulting in a greater amount of precipitate in parallel with a high production of H_2 bubbles gas then a better turbidity removal was observed.
- At 25V, in spite of the high current density, we note a low turbidity removal which can be interpreted by the noted increase of water temperature, which can be explained by the close distance between the two electrodes that affected the destabilization of the ionic links formed between the pollutant and the coagulant.

From these results, we deduced that the optimum condition for the treatment was 20V.

2.1.2. Influence of the mucilage concentration on the turbidity removal efficiency

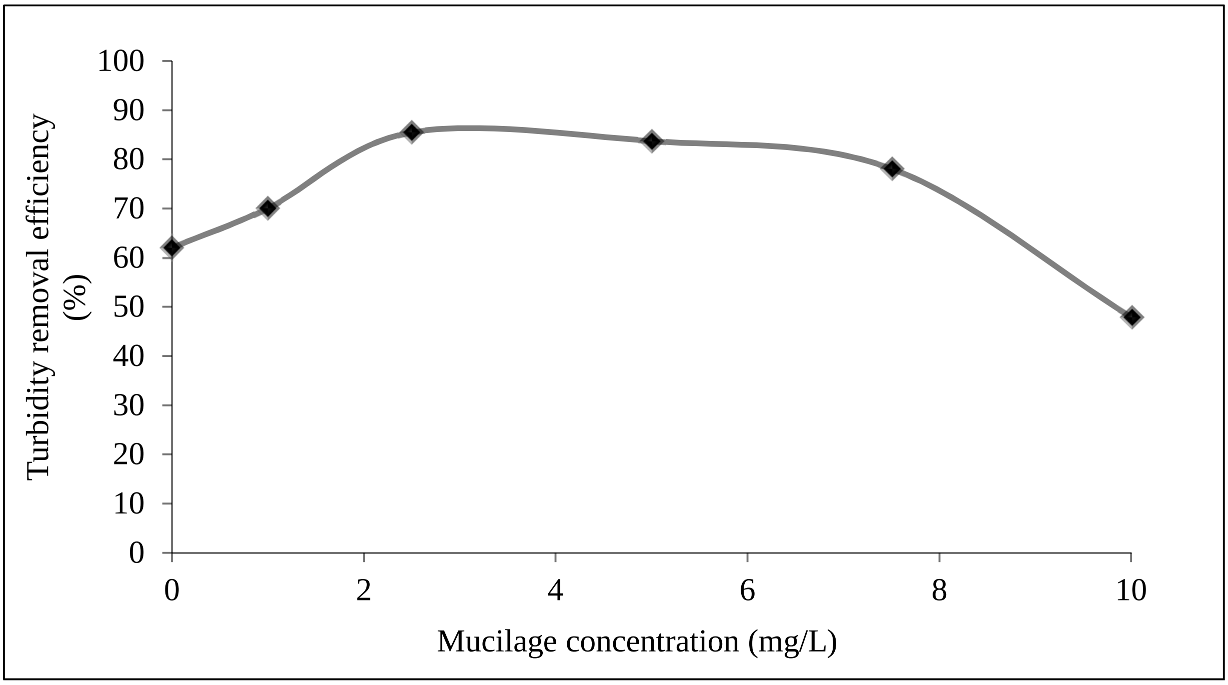

Figure (20): Effect of initial concentration of OFI mucilage on turbidity removal efficiency: initial silica gel concentration C_0 = 300 mg/L, initial pH = 7.67, inter-electrode distance d = 1 cm, conductivity k = 1.31 mS/cm, run time t = 20min, voltage U =20 V.

To study the effect of OFI mucilage on turbidity removal efficiency various OFI mucilage concentration ranging from 1 to 10 mg/L, were tested. EC–EF was carried out under preselected experimental conditions of: initial pH 7.67, distance between electrodes 1 cm, conductivity was set at 1.31 mS/cm, initial synthetic wastewater concentration and voltage were fixed at 300 mg/L and 20 V, respectively. It can be seen that adding of low amounts of mucilage at a concentration of 1 mg/L, the efficacy of the treatment does not increase significantly and the turbidity remove enhance of 8.07 %. At a concentration of 2.5 mg/L the turbidity removal efficiency increase significantly and reached its peak with 85.5% , which is equivalent to 166.64 mg/L, which means that the initial turbidity was reduced by 3 to 2 fold. In addition, the effectiveness of treatment decrease when concentrations of mucilage are important.

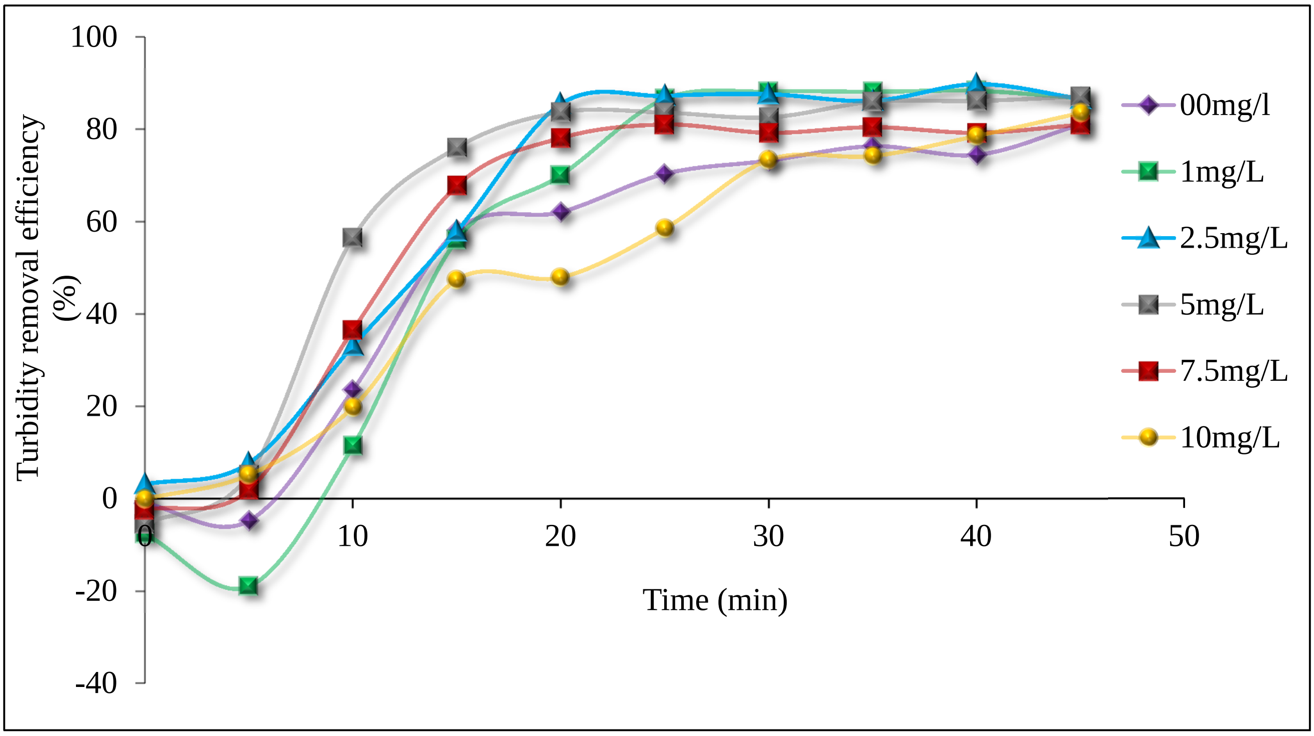

Figure (21): Effect of OFI mucilage on turbidity removal efficiency: initial silica gel concentration $C_0 = 300$ mg/L, initial pH = 7.67, inter-electrode distance d= 1 cm, conductivity k = 1.31 mS/cm, voltage $U = 20$ V.

Figure-21 illustrates the effect of both mucilage concentration and time on turbidity removal efficiency. In initial time (Fig. 21) we note a negative turbidity removal which can be explained by the polymerization of dissolved aluminum in the shape of aluminum hydroxide, thus increasing turbidity (**Khemis and al., 2006**).

It was noted that for almost all tested mucilage concentrations, turbidity removal increases progressively over time and do not change after 20 min run time (Fig. 21). The EC–EF performance attained its maximum levels using 2.5 mg/L mucilage dose when compared to the other tested concentrations. According to this result, the optimum of the mucilage concentration of OFI and run time for pretend wastewater treatment were considered to be 2.5 mg/L and 20 min, respectively. **Miller and al.** (**2008**) found also that small amount (5 mg/L to 15 mg/L) of *Opuntia* spp. cladode powder was efficient to treat 0–125 NTU of synthetic wastewater. The concentration of mucilage used is more important comparing with that of the work of **Adjeroud and al.** (**2015**) with 0.016ml/L of OFI pads juice. These noted difference in coagulant concentration gave different treatment efficacy yields; the OFI pad juice remove 87% ± 0.8 of turbidity removal whereas mucilage was more efficacious with a turbidity removal was in average 93.14%±1.31. **de Carvalho and al.** (**2015**) studied the coupling of EC-EF with banana peel to remove the Methylene Blue, they found that 2 g/L of banana peel concentration helps the treatment to remove 99% of the colorant. Also, this results agree with that obtained by **Ndabigengesere and al. (1995)** who examined the quality of model turbid water treated with *Moringa oleifera* seeds, and **Anastasakis and al.** (**2009**) who studied the assessed of mallow and okra mucilage on the removal of turbidity from synthetic and biologically-treated effluent. Compared to our optimum dose of 2.5 mg/L of OFI mucilage, they used 50 mg/L of *M. oleifera* seeds to reduce turbidity from 105 NTU to 10 NTU (90% reduction) and 12 to 5 mg/L of mallow and okra mucilage to remove turbidity at different time (10, 20 and 30 min), for each time the result were from 81 to 96% and 93 to 96% and 97.3% respectively.

Majdoub and al. (**2001**) isolated the free proteins mucilage fraction and showed that it contains about 20% of charged sugars that can potentially interact with divalent cations. The coagulant activity of OFI mucilage result from his capacity of charge neutralization and a greater water retention capacity (**Sáenz and al., 2004**). Studies proposed using zeta potential measurements and electron microscopy images of formed flocs show that the coagulation mechanisms of *Opuntia spp*. functions principally through adsorption (**Miller and al., 2008**). Furthermore, the coagulation mechanism, **Miller and al. (2008)** tested mucilage sugars independently and in combination were retired that the coagulation activity is predominantly due to galacturonic acid alone or in combination either with arabinose, galactose, or rhamnose. It can be supposed that turbidity removal occurs through physical scavenging of the suspended solids by the dispersed mucilage in water, but the colloidal solids can only be removed by charge neutralization achieved by the electrolysis of aluminum plaques during the electrochemical process. In theory, the aluminum ion generates small flocs, which could be easily attached to the OFI mucilage increasing its coagulation capacity then increasing of the turbidity removal.

Wastewater treatment by electrolysis is a modern treatment process that removes pollutants through electrocoagulation and electroflotation (EC-EF). The key process in electrolysis is the interchange of atoms and ions by the removal or addition of electrons from an external circuit. Electrocoagulation occurs during electrolysis. The anode is involved in rapid adsorption of soluble organic compounds and trapping of colloidal particles that can be easily separated from an aqueous medium by H_2 flotation (**Mollah and al., 2001**) . However, it is probable that the metal ions generated at the anode interact with suspended particles forming small flocs, which could attach to the cactus mucilage increasing its coagulation capacity. The inverse mechanism could also occur. When pH evolution was monitored during investigation of OFI mucilage effect on EC–EF, using increasing cactus dosages, there was no significant effect on the final pH. **Yang and al**. (**2008**) found that cactus powder did not cause a significant effect on final pH of waters when compared to chemical coagulants. They suggest that *Opuntia spp.* operates predominantly through a bridging-coagulation mechanism where solution particulates do not directly contact one another but are bound to a polymer-like material that originates from the cactus species. The presence of galacturonic acid in OFI is one of the major active coagulating agents in plants and therefore, deserves further technical assessment.

Aqueous extracts of dry seeds of *M. oleifera* are one kind of natural macromolecular coagulants (**Agrawal and al., 2011; Duan and Gregory, 2003**). Studies have shown that the water extract of *M. oleifera* seeds has a similar function with aluminium, and it has been recommended as a water treatment coagulant (**Egila and al., 2011**). Whereas Study made in Cuba (**López, 2000**) compared the purifying capacity of the cactus pads mucilage with other traditional agents: $Al_2(SO_4)_3$ and they reported that OFI has a behavior similar to that of the aluminum sulfate in water purifying. The mucilage reduced the oxygen chemical demand (COD) as well, it has also removed metals and fecal coliforms and after the treatment the water did not have any unpleasant odor (**Zhang and al., 2006**).

These conclusions led to the study of **(Egila and al., 2011; Miller and al., 2008)** that reports that cactus has similar properties to seeds of *M oleifera*, so it has the potential to act as a coagulant. The most commonly studied cactus genus for water treatment is *Opuntia*, (**Inglese and al., 1998).** Besides Opuntia, other cactus species including *Cactus latifaria* have also been successfully used as natural coagulants (**Diaz and al., 1999**).

In contrast to chemical coagulants, plant-based natural coagulants are safe (**Asrafuzzaman and al., 2001**), eco-friendly and generally toxic free (**John, 2006**). Natural coagulants have been found to generate not only a much smaller sludge volume of up to five times lower (**Ndabigengesere and al., 1995**) but also with a higher nutritional sludge value. As such, sludge treatment and handling costs are lowered making it a more sustainable option. The raw plant extracts are often available locally and hence, a low cost alternative to chemical coagulants. Since natural coagulants do not consume alkalinity unlike aluminium, pH adjustments can be omitted and this provides extra cost savings.

Natural macromolecular coagulants show bright future and are concerned by many researchers because their abundant source, low price, iniquity, multifunction and biodegradation (**Driscoll and Letterman, 1995)**.

2.1.3. Influence of the pH on the turbidity removal efficiency

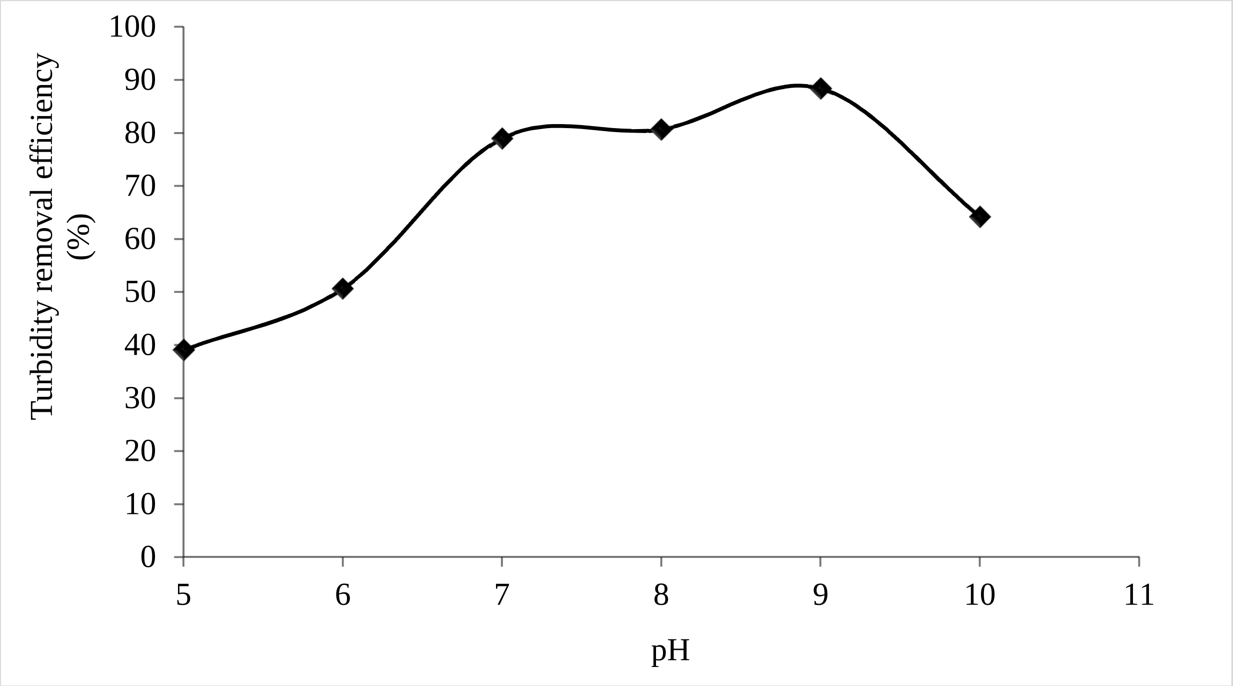

Figure (22): Effect of the pH on turbidity removal efficiency: initial mucilage concentration C_m = 2.5 mg/L, initial silica gel concentration C_0 = 300 mg/L, inter-electrode distance d = 1 cm, run time t = 20 min, conductivity k = 1.31 mS/cm, voltage *U* = 20 V.

Studies have shown that the pH plays an important role as parameter influencing the performance of the electrochemical process (**Chen and al., 2000; Lin and Chen, 1997; Parsa and al., 2011)**. However, pH affects also bubble size (**Srinivasan and al., 1992**).

To investigate the effect of pH on turbidity removal by the EC–EF assisted with OFI mucilage as is shown in Fig.22. Various pH ranging from 5 to 10, were tested. EC–EF was carried out under preselected experimental conditions of: initial OFI mucilage 2.5 mg/L, distance between electrodes 1 cm, conductivity was set at 1.31 mS/cm, initial synthetic wastewater concentration and voltage were fixed at 300 mg/L and 20V, respectively. The sample was adjusted to the desired pH by adding the appropriate amount of 1M NaOH or HCl.

The turbidity removal efficiency reached at its maximum until pH 9 with value of 88.39% which is equivalent to 74.69 ± 4.26 mg/L, this means that initial turbidity was, reduce by 3 to 4 fold. At pH 10, the remove turbidity start to decrease.

Two main mechanisms are generally considered to explain pollution removal: precipitation at pH lower than 4 and adsorption at higher pH. The adsorption may proceed on $Al(OH)_3$ or on the monomeric $Al(OH)^{4-}$ anion. The formation of $Al(OH)_{3(s)}$ is optimal at 4 to 9 of pH range.

According to **Parsa and al. (2011)**, the pH affects $Al(OH)_3$ stability in the solution, the Fig. 23. demonstrates different forms of $Al(OH)_3$ relative to the pH and concentration of Al^{3+} ions **Jiang and al.** (**2002**). In the high and low pH, $Al(OH)_3$ is in its charged form and is soluble in water, hence, cannot be used for EC. But in neutral pH, $Al(OH)_3$ is stable and insoluble in the water and available for pollutant adsorption from water (**Parsa and al.,2011)**.

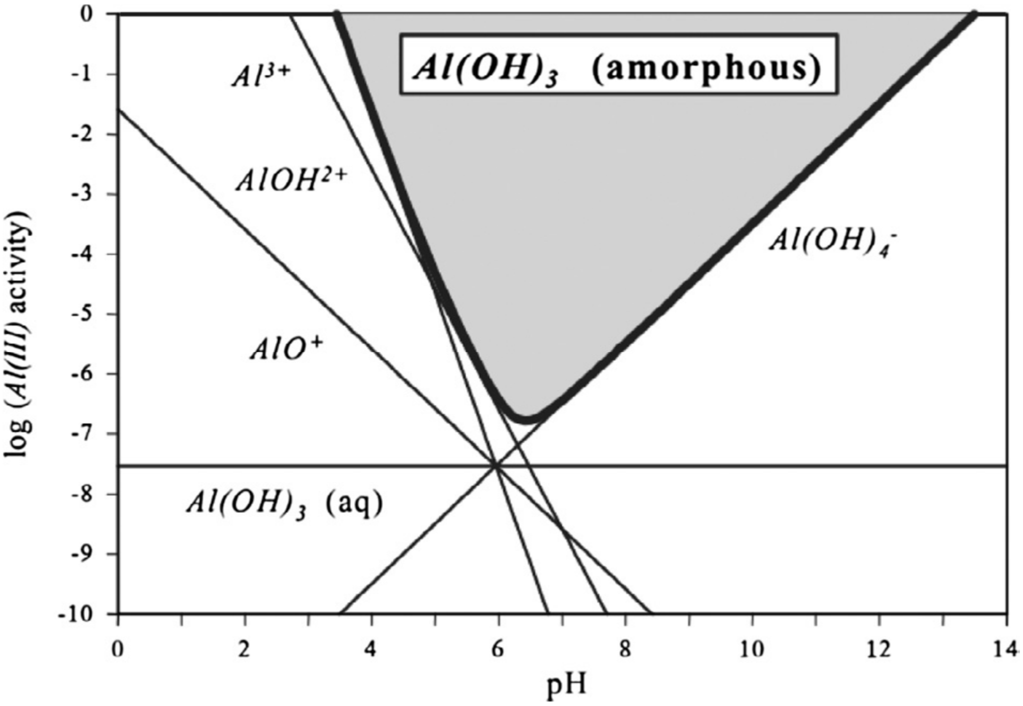

Figure (23): Activity–pH diagram for Al^{3+} species in equilibrium with $Al(OH)_3$ (amorphous) (**Yilmaz and al., 2007**).

According to Figure 23, one can noted two principally reaction, precipitation and adsorption

- Precipitation, they spread out pH < 3, were the cationic monomeric species Al^{3+} and $Al(OH)_2^+$predominate; the trivalent form Al^{3+} becomes negligible in less acid medium (pH > 6).

- Adsorption, they unroll at *pH > 4* were ions generated by the electrode Al^{3+} and OH^- react to from the various. monomeric species such as $Al(OH)_2^+$, $Al(OH)_2^{2+}$ and polymeric species such as $Al_6(OH)_{15}^{3+}$, $Al_7(OH)_{17}^{4+}$ and $Al_{13}(OH)_{34}^{5+}$ that finally transform into insoluble amorphous $Al(OH)_{3(s)}$ through complex polymerization/ precipitation kinetics (**Kobya and al., 2006; Merzouk and al., 2009**).

At *pH > 10*: The monomeric $Al(OH)_4^-$ anion increase at the expense of amphoteric $Al(OH)_{3(s)}$ according to the following reaction (Rq.8), which causes a slight decrease in final pH by consuming OH^- ions.

$$Al(OH)_3 + OH^- \leftrightarrow Al(OH)_4^- \qquad (8)$$

The cathode may be chemically attacked by OH^- ion generated together with the hydrogen ghazis H_2 at high pH values Reaction (9).

$$2Al + H_2O + OH^- \leftrightarrow 2Al(OH)_4^- + 3H_2O \qquad (9)$$

Vila and al. (**2000**) reported that hydrogen bubbles are known to be the smallest about neutral pH while the size of oxygen bubbles increases with pH. Thus, pH may be adjusted in a range that allows equilibrium between best coagulation and best flotation (**Merzouk and al., 2009**).

In addition, **Llerena and al.** (**1996**) found that the recovery of sphalerite was optimal at pH between 3 and 4 using buffer solution. They also documented that within this pH range, the hydrogen bubbles were the smallest, about 16 ± 2 μm. At pH of 6, the mean hydrogen bubbles diameter was 27 μm whereas at pH of 2, the hydrogen bubbles were about 23 μm (**Chen and al., 2010**). Typical bubble sizes in electrocoagulation always fall in the range of 20–70 μm they are far smaller than those observed in conventional air-assisted flotation (**Adhoum and al., 2004**).

Some studies reported in the literature claim that controlling pH in iron-based electrocoagulation processes leads to higher efficiencies (**Secula and al., 2012**). However, the mechanism of electrocoagulation depends mainly on the nature of the pollutant (**Khemis and al., 2006**). **Secula and al.** (**2011**) found that the addition of acid solution to control the pH value of the treated solution in fact limits the development of the flocs generated.

There is a liaison between pH and OFI coagulation activity. Its coagulation ability increases at alkaline pH (**Miller and al., 2008; Nougbodé and al., 2013**). **Torres and Carpinteyro-Urban** (**2012**) observed that at pH 10, OFI mucilage is efficient in removing COD. Also, **Zhang and al.** (**2006**) have reported that *Opuntia spp.* is most effective at pH 10 and is least effective at pH 6. **Adjeroud and al**. (**2015**) have found that the EC-EF treatment assisted by OFI pad juice aid to improve the turbidity removal efficiency at pH 8.2. However, The electrolytes decrease repulsion between particles, and facilitate coagulation that occurs at pH 8 (**Miller and al., 2008**).

So in this study, the initial pH was fixed at 9 to maximize turbidity removal.

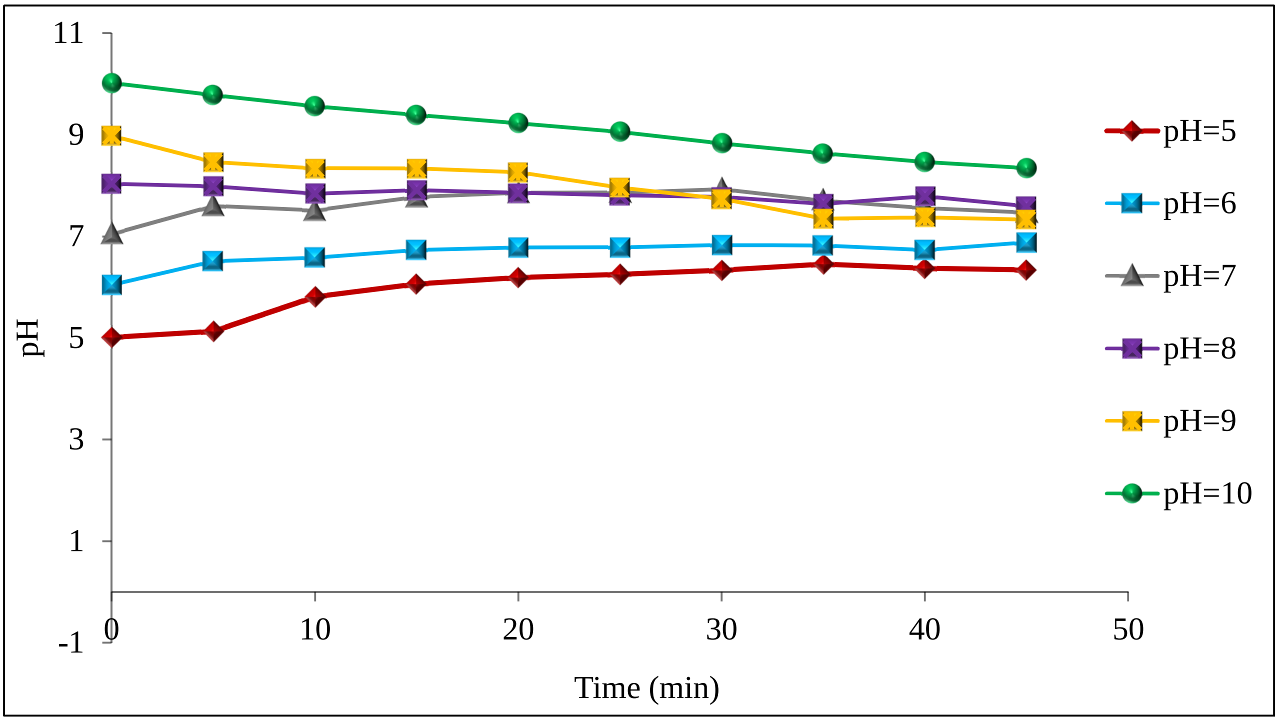

Figure (24): Evolution of the pH values during EC-EF for different values of initial pH : initial mucilage concentration C_m = 2.5 mg/L, initial silica gel concentration C_0 = 300 mg/L, inter-electrode distance d = 1 cm, conductivity k = 1.31 mS/cm, voltage *U* = 20 V.

The figure (Fig.24) showed the pH values according to time during the treatment by EC-EF assisted by OFI mucilage, they demonstrate that pH values tends towards naturalness whatever the initial pH of solution. The ability to neutralize the pH of wastewater is a characteristic of EC-EF that it is showing by several authors (**Adjeroud and al., 2015; Chen and al., 2000; Kobya and al., 2006; Merzouk and al., 2009**). When influent pH is acidic, effluent pH value rises, and when influent pH is alkaline, effluent pH value drops (**Chen and al., 2000**). Thus, the pH value in effluent will be brought closer to neutral, where effective coagulation has been reported. That can be explained by balance production between (OH^-) and their consumption, preventing high changes in pH (**Chen , 2004; Chen and al., 2000**), wich is an advantageous for wastewater treatments.

2.1.4. Influence of the conductivity on the turbidity removal efficiency

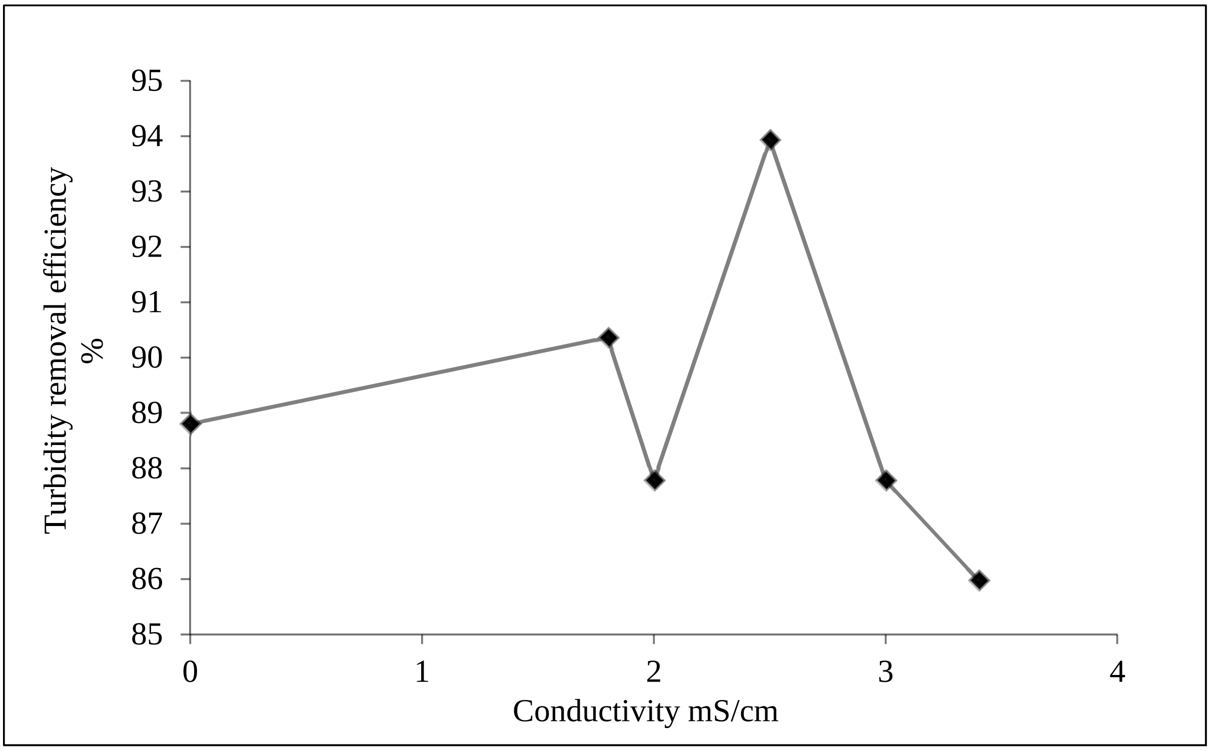

Figure (25): Evolution of the conductivity values during EC-EF for different values of initial conductivity: initial mucilage concentration C_m = 2.5 mg/L, initial silica gel concentration C_0 = 300 mg/L, inter-electrode distance d = 1 cm, pH = 9, run time t = 20 min, voltage *U* = 20 V.

The figure-25. showed the evolution of efficacy turbidity remove according to the conductivity values during the EC-EF assisted by OFI mucilage. The conductivity of the wastewater model was adjusted by adding appropriate volumes of 1M NaCl solution. The conductivity effect was investigated between 1.8 and 3.4 mS/cm, during the treatment using the following experimental conditions: 2.5 mg/L mucilage concentration, initial pH 9, voltage 20 V, EC–EF run time of 20 min, inter-electrode distance 1 cm.

According to the figure (Fig. 25), we show variations of increase and decrease in the remove of the turbidity that according to the values of the conductivity. It is noted that the elimination of turbidity reaches in its efficacy at 2.5 mS/cm with a value of 93.93%, which is equivalent to 43.19 mg/L, which means that the initial turbidity was reduced by 12 to 13 fold. The turbidity removal became to diminish at a high value of conductivity, which are 2.8, and 3.4 mS/cm with value even to 87.78 and 85.98% respectively.

Thus result are similar to that obtained by **Merzouk and al**. (**2009**) how found the efficacy value of conductivity in average of 2.1mS/cm. **Adjeroud and al.** (**2015**) in their study optimization of the effect of conductivity on the OFI pads juice was found in average of 2.7 mS/cm.

In the EC–EF treatment, the conductivity was affects by the cell voltage *U* and consumption of electrical energy. Electrolyte addition employed to increase the conductivity is known to reduce the ohmic resistance of the wastewater to be treated and hence the cell voltage U at constant current density (**Bayramoglu and al., 2004; Daneshvar and al., 2006, Kobya and al., 2006**).

In addition, the energy consumption will decrease since it depends on cell voltage *U* and current intensity *I*. The use of NaCl to increase conductivity has other benefits, chloride ions could significantly reduce the adverse effects of other anions, such as $HCO_3^-$and as SO_4^{2-} (**Chen, 2004**). Carbonate ions cause the precipitation of Ca^{2+} ion that forms an insulating layer on the surface of the cathode increasing the ohmic resistance of the electrochemical cell (**Chen and al;, 2002; Daneshavar and al., 2006**).

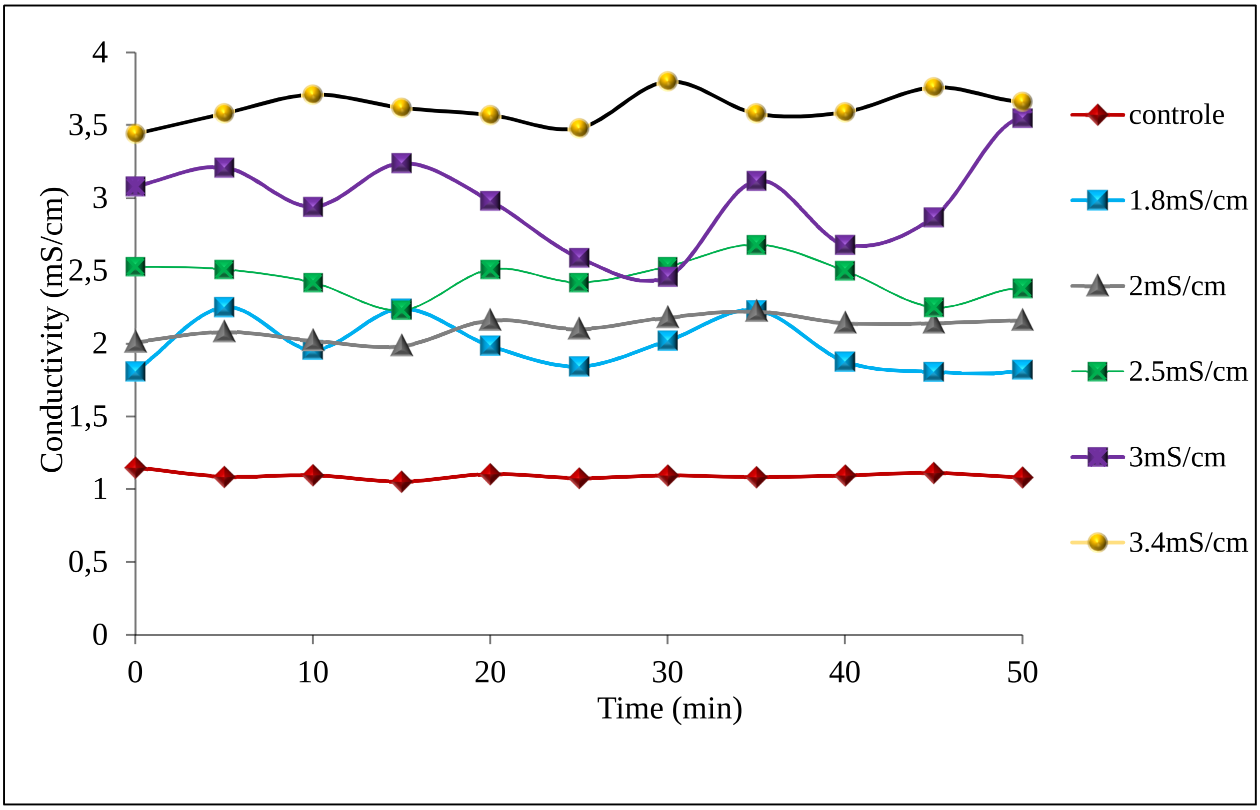

Figure (26): Evolution of the Conductivity values during EC-EF for different values of initial pH : initial mucilage concentration C_m = 2.5 mg/L, initial silica gel concentration C_0 = 300 mg/L, inter-electrode distance d = 1 cm, pH = 9, voltage *U* = 20 V.

The figure above show an unchanged of values of conductivity for each conductivity used and they rest almost stable in each one during the treatment. This result is similar showed by (**Adjeroud and al., 2015; Merzouk and al., 2008**).

The pH value of treated solution increases with the addition of NaCl, which is due to the increase of the solution conductivity and thus to the increase of hydroxide ions concentration. In addition, **Chen and al.** (**2000**) reported that Cl^- or SO_4^{2-} can exchange partly with HO^- in metal hydroxides to free hydroxyl ions, which also causes a pH increase.

Moreover, **Lee and Pyun** (**1999**) reported that the addition of Cl^- ions into solutions lead to an increase of the anodic dissolution rate of the sacrificial electrode, either by the incorporation of Cl^- to the oxide film or by the participation of Cl^- in the metal dissolution reaction.

Therefore, the addition of NaCl to the wastewater is a better choice for increasing the performance of the electrocoagulation process. However, as discussed the decolorization rate decreases when more NaCl was added. Hence, the excessive amount of chloride ions in solution is unfavorable to the coagulation. **Wang and al.** (**2009**) explained this phenomenon by assuming the chloride ions form transitory compounds with the metal hydroxides generated into solution. Moreover, chloride ions might be discharged at the anode and generate Cl_2 that is dissolved into solution and chemically converted to ClO^- which can further oxidize the pollutants (**Wang and al., 2009**).

3. Optimization using Box-Behnken Design (BBD)

3.1. Modeling and fitting the model using Response surface methodology (RSM)

The Table.X represent the non-coded values of experimental variables and 27 experimental points. Three replicates (6- 9- 21) were used to evaluate the pure error. Factor levels were coded as -1 (low), 0 (central point or middle) and +1 (high), respectively.

To verify the adequacy of the models, the EC–EF treatment assisted by OFI mucilage trials were carried out at the optimal conditions predicted by the RSM and the obtained experimental data were compared to the values predicted by the regression model. Efficiency of the cactus juice assisted EC–EF treatment was evaluated based on the turbidity (TR) measured after the EC–EF process run under the optimum conditions selected by RSM.

Table VIII: Factors and levels for RSM Box–Behnken Design (in coded and uncoded level of four variables) with the observed and predicted response values for turbidity removals (%).

Run	Mucilage (mg/L)	pH	Voltage (V)	Conductivity (mS/cm)		
					Exprimentale	Predicted
1	5 (+1)	7 (-1)	20 (0)	2.5 (0)	81.26±4.82	80.94
2	5 (+1)	10 (+1)	20 (0)	2.5 (0)	89.5±1.69	89.26
3	3 (0)	10 (+1)	20 (0)	3 (+1)	90.9±1.49	90.88
4	1.5 (-1)	8.5 (0)	15 (-1)	2.5 (0)	85.05±1.46	85.44
5	3 (0)	8.5 (0)	25 (+1)	3 (+1)	87.8±1.15	87.47
6	3 (0)	8.5 (0)	20 (0)	2.5 (0)	91±0.48	91.26
7	3 (0)	7 (-1)	15(-1)	2.5 (0)	82.42±1.49	82.38
8	1.5 (-1)	10 (+1)	20 (0)	2.5 (0)	90.41±1.56	90.08
9	3 (0)	8.5 (0)	20 (0)	2.5 (0)	91.4±0.01	91.26
10	1.5 (-1)	8.5 (0)	20 (0)	2 (0)	83±2.47	83.86
11	3 (0)	8.5 (0)	15 (-1)	3 (+1)	88.06±2.06	87.82
12	5 (+1)	8.5 (0)	20 (0)	3 (+1)	82.82±3.4	83.1
13	3 (0)	8.5 (0)	25 (+1)	2 (-1)	88.66±0.96	88.24
14	3 (0)	10 (+1)	20 (0)	2 (-1)	89±0.67	88.59
15	1.5 (-1)	7 (-1)	20 (0)	2.5 (0)	84.57±2.91	84.16
16	1.5 (-1)	8.5 (0)	20 (0)	3 (+1)	89.68±0.23	89.56
17	3 (0)	10 (+1)	25 (+1)	2.5(0)	91±2.04	91.68
18	3 (0)	7 (-1)	20 (0)	2 (-1)	82±0.7	82
19	5 (+1)	8.5 (0)	15 (-1)	2.5 (0)	85.88±0.92	85.78
20	5 (+1)	8.5 (0)	25 (+1)	2.5 (0)	86±0.8	85.59
21	3 (0)	8.5 (0)	20 (0)	2.5 (0)	91.38±0.07	91.26
22	3 (0)	7 (-1)	20 (0)	3 (+1)	82.84±0.49	83.23
23	3 (0)	10 (+1)	15 (-1)	2.5 (0)	90±1.85	90.29
24	3 (0)	8.5 (0)	15 (-1)	2 (-1)	83.85±5.06	83.52
25	5 (+1)	8.5 (0)	20 (0)	2 (-1)	85±1.24	85.77
26	3 (0)	7 (-1)	25 (+1)	2.5 (0)	85±7.45	85.35
27	1.5 (-1)	8.5 (0)	25	2.5 (0)	89.9±2.32	89.98

Table IX: Estimated regression coefficient for the quadratic polynomial model and the analysis of variance (ANOVA) for the experimental results.

Parameter[a]	**Estimated coefficients**	**Standard error**	**DF**[b]	**Sum of squares**	**F-value**	**Prob>F**
Model Intercept	91.26	0.3	14	293.18	80.2	
B_0	91.26	0.3	1	293.18	80.2	<0.0001
Linear						
X_1 (Mucilage)	-1.01	0.15	1	12.3	47.11	<0.0001
X_2 (pH)	+3.56	0.15	1	152.08	582.4	<0.0001
X_3 (Voltage)	+1.09	0.15	1	14.3	54.76	<0.0001
X_4 (Conductivity)	+0.88	0.15	1	9.35	35.79	<0.0001
Quadratic						
X_1^2	-2.94	0.22	1	46.03	176.29	<0.0001
X_2^2	-2.21	0.22	1	26.03	99.68	<0.0001
X_3^2	-1.62	0.22	1	13.98	53.55	<0.0001
X_4^2	-2.87	0.22	1	43.94	168.28	<0.0001
I***nteraction***						
X_1X_2	+0.6	0.26	1	1.44	5.51	0.0368
X_1X_3	-1.18	0.26	1	5.59	21.42	0.0006
X_1X_4	-2.22	0.26	1	19.62	75.15	<0.0001
X_2X_3	-0.39	0.26	1	0.62	2.39	0.1481
X_2X_4	+0.27	0.26	1	0.28	1.08	0.3201
X_3X_4	-1.27	0.26	1	6.43	24.61	0.0003
Lack of Fit			10	3.03	5.97	0.1519
Pure Error			2	0.1		
Residual			12	3.13		
R^2					0.9894	
$R^2_{adjusted}$					0.9771	
C.V. %	0.59					
RMSE	0.5110					
Core total			26	296.32		

[a] Coefficient refer to the general model; [b] Degree of freedom.

Based on the preliminary results reported in Table X, the following parameter ranges: 1–5 mg/L, 7–10, e 15-25 V and 2–3 mS/cm were selected for initial OFI mucilage concentration, initial pH, initial voltage and initial conductivity respectively, in order to achieve RSM optimization tests.

Turbidity removal efficiencies obtained in the trials of the BBD. Confirming the results of the preliminary trials, the turbidity removal efficiency reached a maximum level when OFI mucilage concentration, pH, voltage and conductivity were set at medium levels (0 coded values) (Table X).

The regression coefficients of the intercept, linear, quadratic and interaction terms of the model were calculated using the least square technique (**Zhang and al., 2013**) and are given in Table XI. It was shown that two linear parameters, mucilage concentration (X_1), pH (X_2), voltage (X_3), conductivity (X_4) and their quadratic parameters were highly significant at the level of ($P < 0.01$), whereas all the interaction parameters ($X_1X_3, X\ _1X_4$, X_3X_4) were highly significant at the level of ($P < 0.01$), the interaction (X_1X_2) was also significant at the level of ($P < 0.05$) but the interactions (X_2X_3, X_2X_4) were insignificant ($p > 0.05$) (**Dahmoune and al., 2013**). Discounting the non-significant parameters ($p > 0.05$), the final mathematical equation predictive correlating the turbidity removal efficiency with EC-EF treatment parameters is given below (Eq. (10)):

$$\boxed{\begin{array}{l} Y(TR)=91.26-1.01X_1+3.56X_2+1.09X_3+0.88X_4+0.60X_1X_2-1.18X_1X_3-2.22X_1X_4-0.39X_2X_3 \\ +0.27X_2X_4-1.27X_3X_4-2.94X_1^2-2.21X_2^2-1.62X_3^2-2.87X_4^2. \end{array}} \quad (10)$$

The analysis of variance (ANOVA) for the experimental results given in Table (XI) shows that model is at *F-value* of 80.20 implies that the model is significant. There is only 0.01% chance that a‟Mode *F-value*″ this large could occur due to the noise.

The determination coefficient R^2 was 0.98, which implied that the sample variation of 98.9% for the OFI mucilage additional in EC-EF efficiency were attributed to the independent variables, and only 1.1% of the total variations cannot be explained by the model. However, a large value of R^2 does not always mean that the regression model is good one (**Karazhiyan and al., 2011**). In a good statistical model, $R^2_{\mathbf{adjusted}}$ should be comparable to R^2 (**Zhang and al., 2011**). As in Table (XI) $R^2_{\mathbf{adjusted}}$ and R^2 values for the model did not differ greatly.

The Lack of Fit ‟*F-value*″ of 5.97 implies the Lack of it is not significant relative to the pure error ($p > 0.05$). There is 15.19% chance that Lack of Fit ‟*F-value*″ this large could occur due noise. The insignificant of Lack of Fit ‟*F-value*″ confirming the validity of the model.

The value of coefficient of variation (C.V.%) was 0.59% and‶Adequate Precision″ratio 28.218 suggest that the model was reliable and reproducible agreeing previous reports (**Chen and al., 2012**). In general, a C.V, a C.V. higher than 10% indicates that variation in the mean value is high and does not satisfactorily develop an adequate response model (adequate signal for the model) (**Karazhiyan et al., 2011**). The results indicated an adequate signal for the model suggesting that it could work well for water treatment by EC-EF technique assisted by OFI mucilage used in this study.

3.2. Analysis of response surface methodology (RSM) and contour plots

The effects of the independent variables and their mutual interactions on the turbidity remove efficiency can be visualized on the three dimensional response surface plots and two dimensions contour plots shown in Fig.28 and Fig.29, respectively. The plots were generated by plotting the response using the z-axis against two independent variables while keeping the other two independent variables at their zero level (**Hayat and al., 2009**). Consequently, the quadratic experimental model had a stationary point, and the predictive turbidity removal efficiency was the maximal value in the stationary point (**Pan and al., 2012**). Each 3D plot represents the number of combinations of the two-test variable. 3D response surface and 2D contour plots are the graphical representations of regression equation and are very useful to judge the relationship between independent and dependent variables. Different shapes of the contour plots indicate whether the mutual interactions between the variables are significant or not. Circular contour plot means the interactions between the corresponding variables are negligible, while elliptical contour suggests the interactions between the corresponding variables are significant (**Liu and al., 2013**).

Figure a-b and c: depict the interaction between the amount of OFI mucilage concentration and each of the three other factors (voltage, pH and Conductivity) on the EC- EF treatment. They shows that the recovery of turbidity removal efficiency increased with increase of the voltage and conductivity at their medium values, also increase with the increase in pH and decreasing the OFI mucilage concentration.

Figure d: the turbidity removal efficiency increased gradually to reach its maximal value with increasing pH and voltage value.

Figure e:. the recovery of turbidity removal efficiency its threshold maximum reached its maximum value with increasing pH and conductivity value.

Figure f: the recovery of turbidity removal efficiency were increasing at the medium values of conductivity and voltage.

As shown in table XI, the turbidity removal efficiency principally depends on the OFI mucilage concentration, pH, voltage and conductivity as them linear and quadratic effects were highly significant ($p<0.01$). The interaction effects between OFI mucilage concentration-pH was significant ($p<0.05$). The interaction OFI mucilage concentration with voltage and conductivity voltage also the interaction voltage-conductivity were highly significant ($p<0.01$).

Generally, these results found are close to that esteemed by the preliminary study which show that the increase in the pH values, conductivity and the voltage in certain limit led to the reduction in the efficacy of the treatment. The same for a concentration of OFI mucilage superior of 2.5mg/L lead also to decrease the turbidity removal efficiency.

4. Validation and verification of predictive model

The results experiments that were performed at the optimized turbidity removal efficiency using EC-EF treatment obtained using RSM had the following critical values:

-OFI mucilage concentration :	2.5 mg/L ;	**-Conductivity:**	2.61 mS/cm;
-pH:	9.65;	**-Voltage:**	21.2 V.

The appropriateness of the model equation for predicting the optimum response values was tested using the selected optimal conditions. The predicted turbidity removal efficiency value was 92.96% ± 0.01 which, was consistent with the practical turbidity removal value of 93.14 ± 1.31. The predicted values were in close agreement with the experimental values (Table X) and were found to be not significantly different at $p > 0.05$ using a paired t-test (**Houssain and al., 2012**). The predicted response values are nearly equal to the experimental data. The normal probability at residuals indicated no abnormality in the methodology adopted. The strong agreement between the real and predicted results confirmed that the response of regression model was adequate to reflect the expected optimization (**Zhang and al., 2013**).

Finally, figure- 27 show the comparison of the turbidity removal efficiency values obtained by RSM optimized EC–EF assisted by OFI mucilage concentration and conventional EC-EF. The turbidity that removal efficiency with conventional EC–EF (62.02%) and those EC–EF assisted by OFI mucilage 93.14% ± 1.31 shows that the optimized process by RSM allows an enhancement of the electrochemical process by 30.94%.

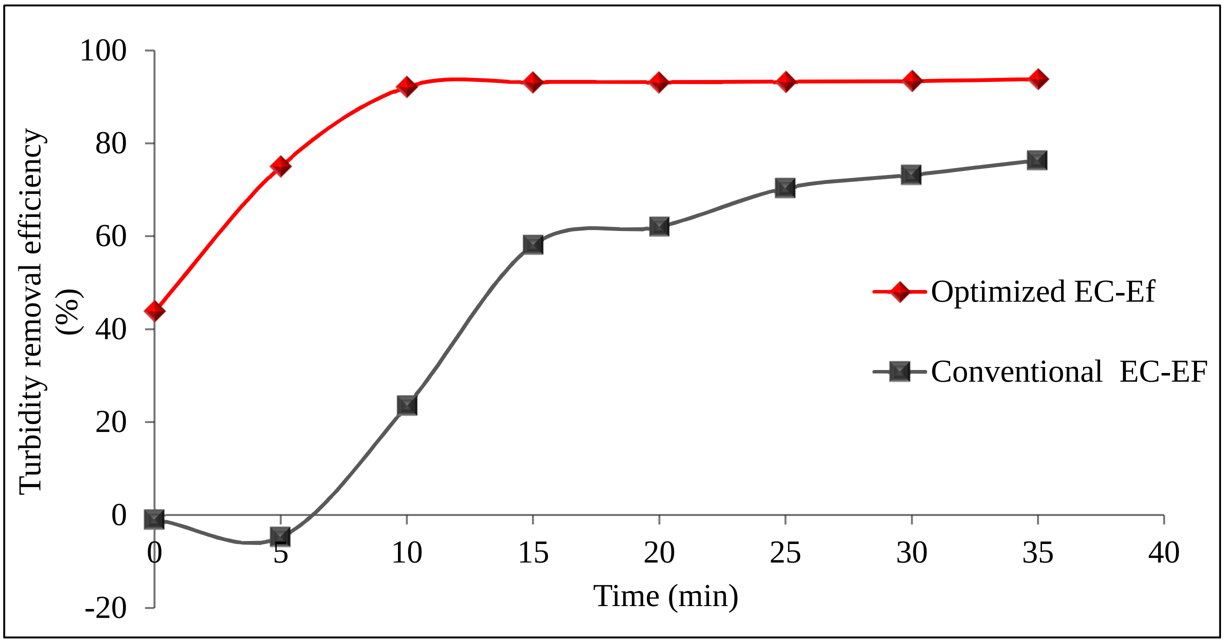

Figure (27): effect of RSM optimization on turbidity removal efficiency. Conventional EC-EF: OFI mucilage C_m = 00 mg/L, pH = 7.67, conductivity k = 1.3 mS/cm, voltage U = 21.2V. Optimized EC-EF OFI mucilage C_m = 2.5 mg/L, pH = 9.65, Conductivity k= 2.61 mS/cm, voltage U = 21.2V. Both EC-EF are realized at initial concentration of silica gel C_0 = 300 mg/L, inter-electrode distance d = 1cm.

The figure-27 demonstrate the comparison between the two methods on the abatement of the turbidity, conventional EC-EF and Optimized EC-EF. According to the result in the table X and XI, the figure above display that the EC-EF assisted by OFI mucilage remove turbidity at 93.14% when the conventional EC-EF with 62.02%. Then the Optimized RSM process allows an improvement of the electrochemical process by 30.94%.

This result is similar to that found by **Adjeroud and al.** (**2015**) on the amelioration of the treatment by EC-EF additional with OFI pads juice. They found that the optimized EC-EF with added of 0.016mg/L of OFI pads juice enhancement the remove turbidity with 15.1%.

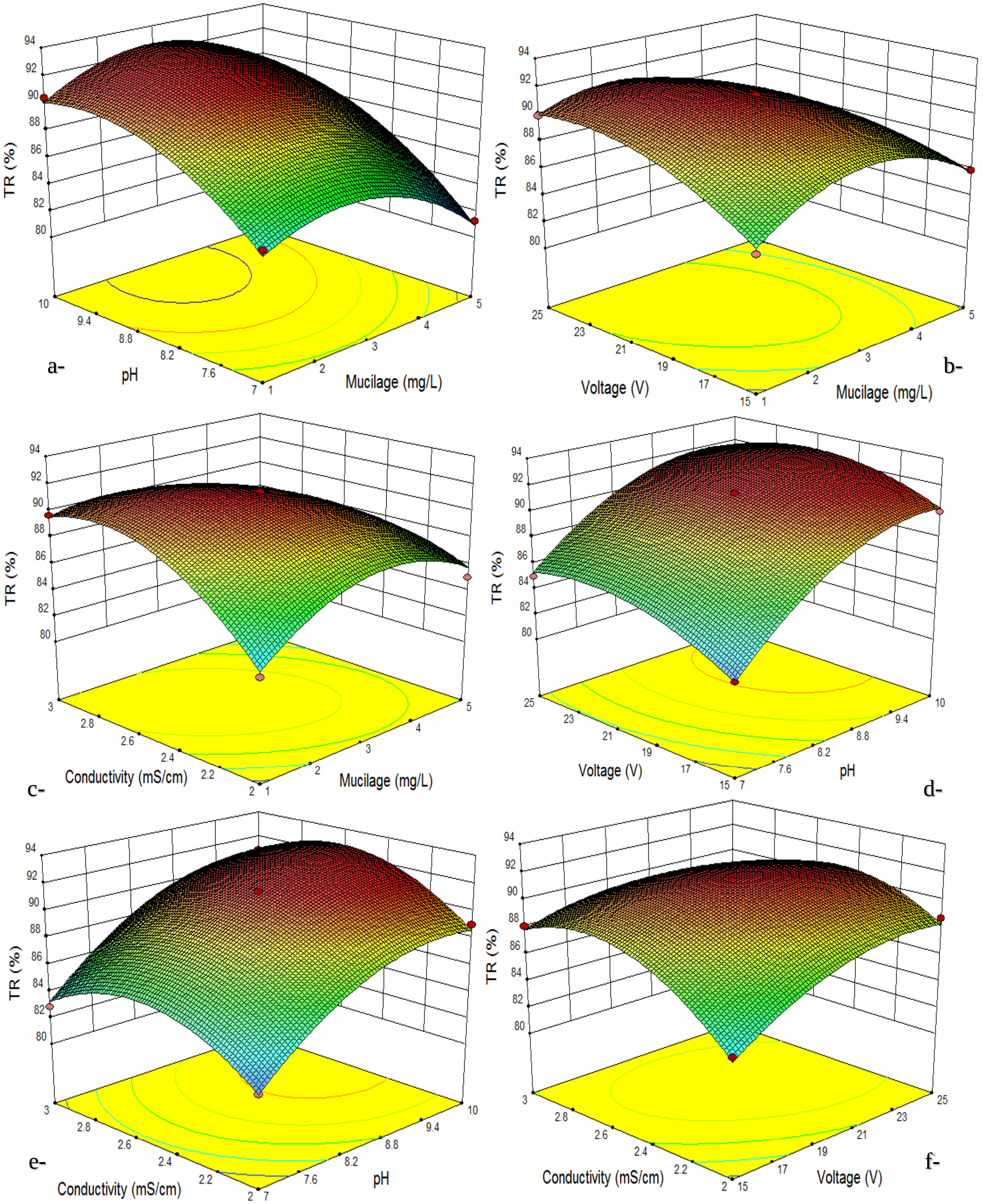

Figure (28): Reponse surface analysis for the EC-EF treatment assisted by OFI mucilage concentration. **(a)** Mucilage and pH; **(b)** Mucilage and voltage; **(c)** Mucilage and conductivity; **(d)** pH and voltage; **(e)** pH and conductivity **(f)** Voltage and conductivity

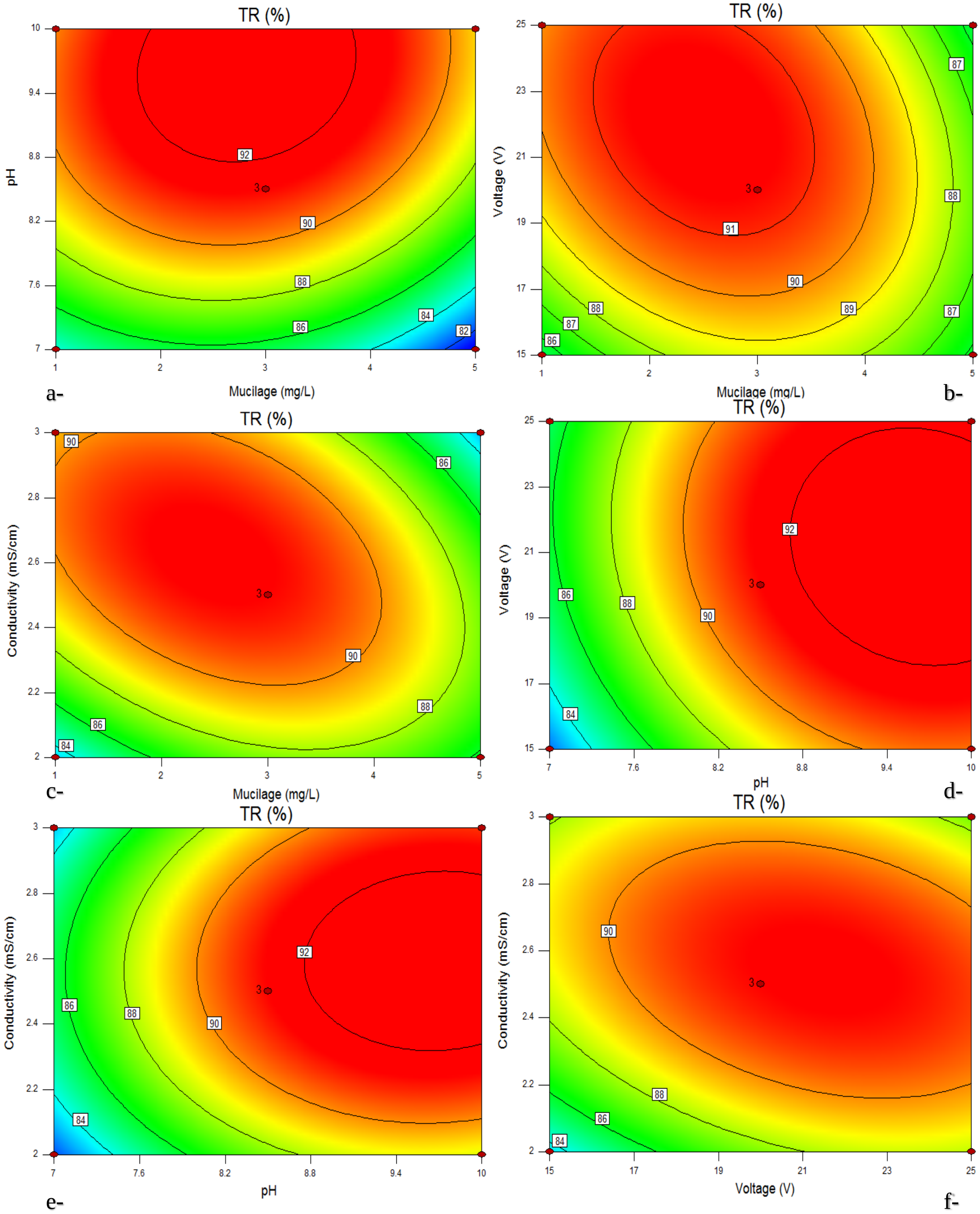

Figure (29): Contour plots analysis for the EC-EF treatment assisted by OFI mucilage concentration. . **(a)** Mucilage and pH; **(b)** Mucilage and voltage; **(c)** Mucilage and conductivity; **(d)** pH and voltage; **(e)** pH and conductivity, **(f)** Voltage and conductivity.

Conclusion

We introduced a biodegradable natural coagulant extract from the prickly pear racket that is mucilage to the EC-EF treatment technique, which already proved its competence, in order to ameliorate its efficiency. The addition of this mucilage improved the efficacy of this method. The orientation towards natural substances helps downplayed the toxicity of chemicals (ferric chloride and aluminum sulfate) on human health and the environment as well as reduces the cost of industrial effluent treatment. In this respect, the results obtained through this work suggest an opportunity for the use of the OFI mucilage as a natural coagulant aid to improve the turbidity removal efficiency of the EC–EF technique.

The response surface methodology was successfully employed to optimize the EC–EF process. Under the optimal operating conditions (initial OFI mucilage concentration C = 2.5 mg/ L, initial pH 9.65, conductivity k = 2.61mS/cm and the voltage = 21.2 Volt), the turbidity removal was enhanced by 30.94% at conditions of run time t = 20 min, initial silica gel concentration C_0 = 300 mg/L and interelectrode distance d = 1 cm. The mathematical model allows optimizing the changing parameters for a good improvement and modification of EC-EF process. However, the standardized equations used in RSM have no methodological background. Consequently, the designed model can only be used within the experimental range and cannot be used for extrapolation. Furthermore, the RSM was useful to investigate the effect of the four studied parameters (OFI mucilage concentration, pH, conductivity and voltage) on the turbidity removal response. The applied second-order polynomial model gave a satisfactory description of the experimental data; it showed that turbidity removal efficiency was affected by the forth-studied parameter.

In summary, this modest work is an good and original alternative because it is a highly effective treatment that reduces the turbidity removal efficiency to the level of 93%, thus this promotes the protection of the environment by reduction of the chemical products that can be added to conventional EC-EF treatment wastewater technique.

This study is certainly incomplete, but is an open door for further works with the same ambitions and perspectives. It would be interesting to complete by:

- Chemical characterization of OFI mucilage (sugars composition, proteins, minerals contents...)
- Application of OFI mucilage assisted EC-EF technique on a real industrial wastewater.
- Elucidation of the mechanism implicated in the interaction between aluminum species and

mucilage, and/or the interaction of the mucilage with suspended silica ionic species in the treatment tank.

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Annex

Annex I

Table I. Solvents and chemicals used for investigations

	Chemicals	**Concentration**
Extraction	-Ethanol	- 95 % (v/v)
pH	-NaOH -HCL	- 1M - 1M
Conductivity	- NaCl	- 1M
Turbidity	-Silica gel	- Woelm Pharma

Table II. Apparatus used for investigation

Apparatus	**Brand**
Precision balance	RADWAG WPS 600/C/2
Spectrophotometer UV-Vis	Spectro Scan 50 (Japan)
Oven	MEMMERT (Germany)
pHmeter	pH211.HANNA Instruments (Romania)
Lyophilizer	Telstar (Spain)
Multiparameter	Extech instruments (French)

Annex II

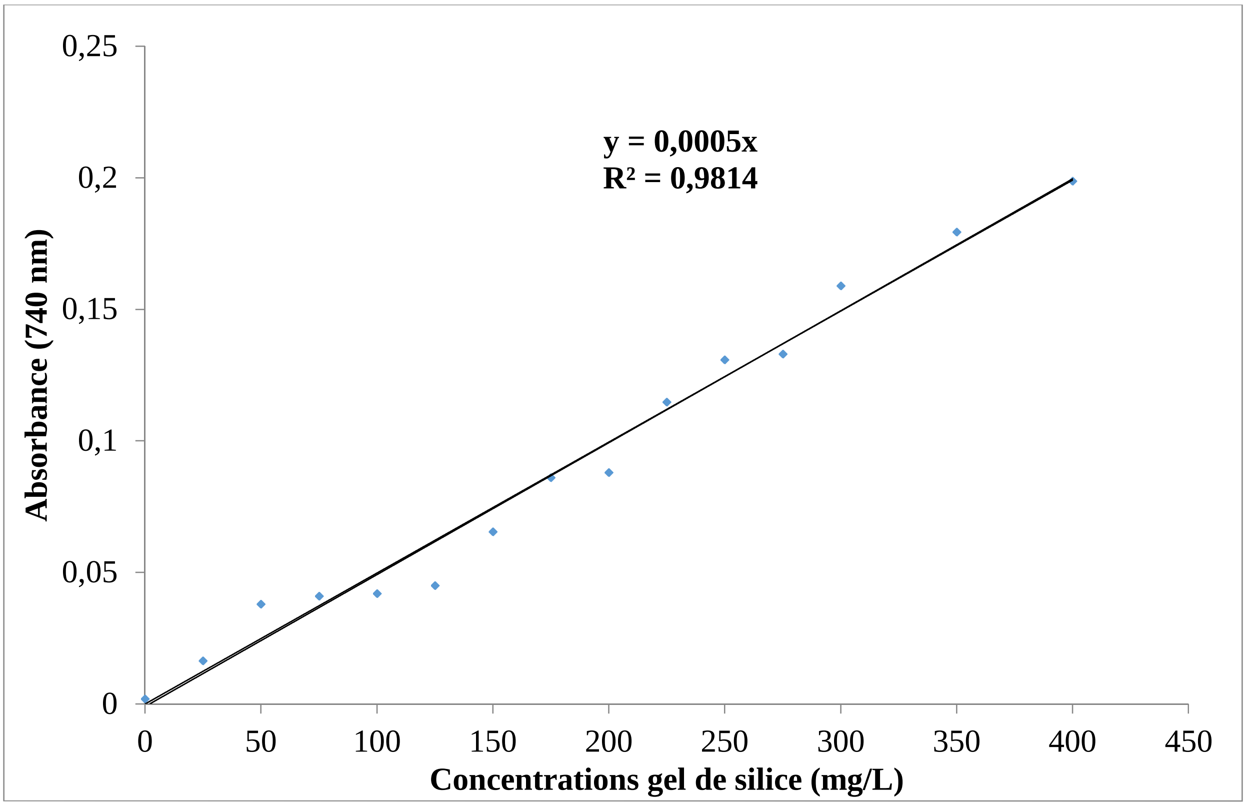

Figure: Calibration curve for the turbidity measurement.

Abstract

The objective of this document is to optimize a water treatment method: Electrocoagulation-electroflotation assisted by *Opuntia ficus indica* (OFI) mucilage, as a natural coagulant, using response surface methodology (RSM). Four parameters (mucilage concentration, conductivity, pH and voltage) that may influence the EC-EF treatment performance were studied. After a preliminary study, intervals to be used for RSM optimization were set for each parameter. The results obtained after RSM optimization for initial OFI mucilage concentration, initial pH, initial voltage and initial conductivity were 2.5 mg/l, 9.65, 21.2 V and 2.61 mS/cm, respectively. Under these optimized conditions, the predict value of the turbidity removal efficiency (TR%) was 92.96%, this predicted value was close to the experimental value of 93.14%±1.31. Compared to the conventional EC-EF (without OFI mucilage addition), which had a TR% value of 62.02%±1.45, optimization of EC-EF assisted with OFI mucilage concentration enable a TR% enhancement of 30.94%. These results indicate suitability and validation of the employed model and the success of RSM in optimizing the EC-EF treatment conditions.

Keywords: Mucilage, *Opuntia ficus indica*, Natural coagulant, Electrocoagulation-electroflotation (EC-EF), Response surface methodology(RSM).

Résumé

L'objectif de ce document est d'optimiser une méthode de traitement de l'eau: électrocoagulation-électroflottation assisté par les mucilage d'*Opuntia ficus indica* (OFI), comme coagulant naturel, en utilisant la méthodologie de surface de réponse (RSM). Quatre paramètres (concentration de mucilage, conductivité, de pH et de tension) qui peuvent influencer la performance de traitement EC-EF ont été étudiés. Après une étude préliminaire, des intervalles à utiliser pour l'optimisation RSM ont été établis pour chaque paramètre. Les résultats obtenus après optimisation par RSM dont pour la concentration initiale de mucilage d'OFI, pH initial, la tension initiale et conductivité initiale ont été de 2,5 mg / l, 9,65, 21,2 V et 2,61 mS / cm, respectivement. Dans ces conditions optimisées, la valeur prédite de l'efficacité d'élimination de la turbidité (TR%) était de 92,96%, cette valeur prédite était proche de la valeur expérimentale trouvée qui été de 93.14% ± 1,31. Par rapport à l'EC-EF conventionnelle (sans ajout de mucilage d'OFI), dont la valeur de TR% trouvée été de 62.02% ± 1,45, l'optimisation de l'EC-EF assisté par le mucilage de la raquette d'OFI permet une amélioration TR% de 30,94%. Ces résultats indiquent l'aptitude et la validation du modèle utilisé et le succès de RSM en optimisant les conditions de traitement par EC-EF.

Mots-clés: Mucilage, Opuntia ficus indica, coagulant naturel, électrocoagulation-électroflottation (EC-EF), méthodologie de surface de réponse (RSM).

الملخص

الهدف من هذه الوثيقة هو تحسين طريقة معالجة المياه بطريقة طفو الالكترونات بإضافة معقد سكري للصبار كمخثر طبيعي من خلال منهجية السطح بعد الدراسة الاولية لتأثير بعض المعايير عل نشاط المعقد السكرى والمعالجة بطريقة طفو الالكترونات مثل (تركيز المعقد السكرى، الموصلية، التركيز الحامضي و المقاومة). قمنا بتحديد مجال أولي لكل من المعايير حيث كان تركيز المعقد السكرى من 1 الى5 مغ, الموصلية 2 الى 3مس/سم, التركيز الحامضي 7 الى10 و المقاومة 15 الى25 فولط. النتائج المتحصل عليها بعد التحسين بالمنهج السطحي للتركيز الاولي للمعقد السكرى كان: 2.5مغ/ل، التركيز الحامضي الاولي: 9.65، الموصلية الاولية: 2.61مس/سم والمقاومة: 21.2 فولط. في هذه الشروط المحسنة، القيمة الفعالة لإزالة العكارة المتنبئة كانت 92.96% هذه القيمة المتنبئة كانت قريبة من القيمة التجريبية التي 93.14 ±1.31 % مقارنة مع الطريقة الكلاسيكية لطفو الالكترونات (أي بدون اضافة المعقد السكرى للصبار) وجدنا ان قيمة العكارة تساوي 62.02% ± 1.45 المعزز بإضافة المعقد للصبار يسمح بتحسين العكارة ب30.94%. هذا الناتج يوضح امكانية تقبل النموذج المستخدم و نجاح المنهج السطحي بتحسين شروط العلاج بطفو الالكترونات.

كلمات المفتاح: معقد سكري,الصبارالهندي, مختر طبيعي, طفو الالكترونات,منهجية السطح