Issue 5 volume 2, March-April 2015
ISSN 2249-9954

Cleaning and Protecting Demineralization Plant from Organic matter/Iron Fouling

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ABSTRACT:

In this work the process of water demineralization is operated in the demineralization plant, which contains both cation and anion resin. One of the important problems facing the process is the organic matter fouling of the anion resin. The physicochemical characteristics of the raw inlet water revealed that the inorganic components were almost constant all over the year of test, from January to December 2014. However, the organic matter content and the most probable number (MPN) of coliform were considerably increased in summer months (August) than in winter (January). The problem of resin fouling is matched with the quantity of organic matter in raw water. Separate or mixed solutions of either NaOH , NaCl , or NaOCl, at different concentrations, showed an activity towards the cleaning of anion resin, but to a variable degree. The most potent solution was a mixture of 5% NaOH and 10% NaCl, so it was used to clean the anion resin from organic fouling and 4% HCl for iron organic matter complex. When the cleaning process is applied in washing tank, it achieved a satisfied result that was indicated by the reduction of both rinse time of the resin and the produced effluent conductivity. The number of service cycles of the plant was improved and increased to 90 cycles, similar to that of the new resin; thereby its duration will be increased. Nevertheless the protective mode such as multimedia sand and carbon filter as well as the pretreatment of water before passing through the ion exchange unit improve alsoeach of number and length of unit cycle and reduce rinse time of the unit.

Key word: Organic fouling - Anion resin - esincleaning.

I-INTRODUCTION:

Rivers, lakes and estuarine environments are deteriorated due to unabated discharge of either organic or inorganic pollutants. Microorganisms are among the organic pollutants that change the quality and nature of water (***Davis et al. 1995***). Such organisms like algae, fungi, viruses, bacteria and protozoa are not only present in surface waters, but also in ground one (***Ericsson, B. and Gtrogardh,O.1997, Escobar, I.C et al., 2001***). Water sources may contain different types of bacteria as Pseudomonads, Achromobacteria, Flavobacteria, Micrococci, aerobic and anaerobic sporebears enterobacteria and streptomycetes (***Austin et al., 1996; Goni-Urrizaetal., 1999 and Wong, S.,et al., 2002)***.

Organic matter contaminants contain tannins, tannic, humic and fulvic acid, which block the strong base sites of the resin causing its blockage or fouling (***Collins, C.H. and Lynes, P.M. 1985,Cornelissen, E.R. et al., 2008***. ***And Davis,C.M; et al1995)***. On the other hand, resin fouling by organic matter, derived from vegetation decay which can make complex with iron, present in water, causing iron fouling of resin (***Abbt-Braun,G., et al 2004 and Betz, 1991***). It is worthy to mention that, organic matter and iron fouling of resin stands as a barrier against the process of water demineralization ***(Purolite 2014)***. It causes aggregation of resin particles that reduces the salt splitting capacity and the ion exchange rate; hence the produced demineralized effluent suffers from poor quality and high conductivity. Raw water which comes from Nile is pretreated with alum, as coaggulant, to precipitate some of the suspended organic matters (about 5-10 %) ***(Humbert, et al., 2007 and Morran, J.Y.et al., 2004)***. After precipitation, water passes through a number of sand filters. The filtered water undergoes either chlorination, to be used for drinking, or demineralized to be used in different industrial purposes. Water demineralization is carried out in demineralization plant that is composed of two units; each contains two vessels. The first vessel is packed with, a strong cation exchange resin bed that eliminates the cationic ions from water whereas the second contains a strong anion exchange resin bed that eliminates the anionic ions. This cycle of water demineralization is called (**service cycle**). At the end of each cycle, the demineralization units become exhausted and should be regenerated and recharged using either 4% HC1 or 5% NaOH for both cation and anion resin, respectively. After regeneration, excess acid or alkali must be rinsed by demineralized water. The time consumed in rinsing is called (**rinse time**) and it depends on the quality of the resin, either cleaned or fouled ***(Tan, Y.R., Kilduff, J.E., 2007)***.

It is worthy to mention that, the long rinse time; poor quality and high conductivity of the produced demineralized effluent are the permanent features of resin fouling. Such fouling usually occurs only in anion resin bed causing about 47% reductions in its efficiency. Concerning rinse time, the new anion resin needs 20 mints rinsing, after each regeneration, while the fouled needs more rinse time and consequently more rinse water. Anion resin fouling often begins after 50 service cycles of water demineralization and it is more evident and worried in summer months (July, August) than in winter and autumn. Accordingly, it is very necessary to clean and rejuvenate the fouled resin from the microbial and organic fouling. Traditional methods for cleaning used separate solutions of either NaOH , NaCl or NaOCl ***(Betz, 1991)*** but still these methods don't achieve satisfied results as indicated by the long rinse time.

Consequently, the objective of this study is to choose different cleaning solutions and examined it for reducing and eliminates the growth of these microorganisms in a trial to apply the most powerful one in cleaning, rejuvenating and protecting the fouled resin from deterioration caused by these contaminants. Thereby, the rinse time will be reduced, the quality of the produced effluent will be improved and the duration of the demineralization unit will be increased.

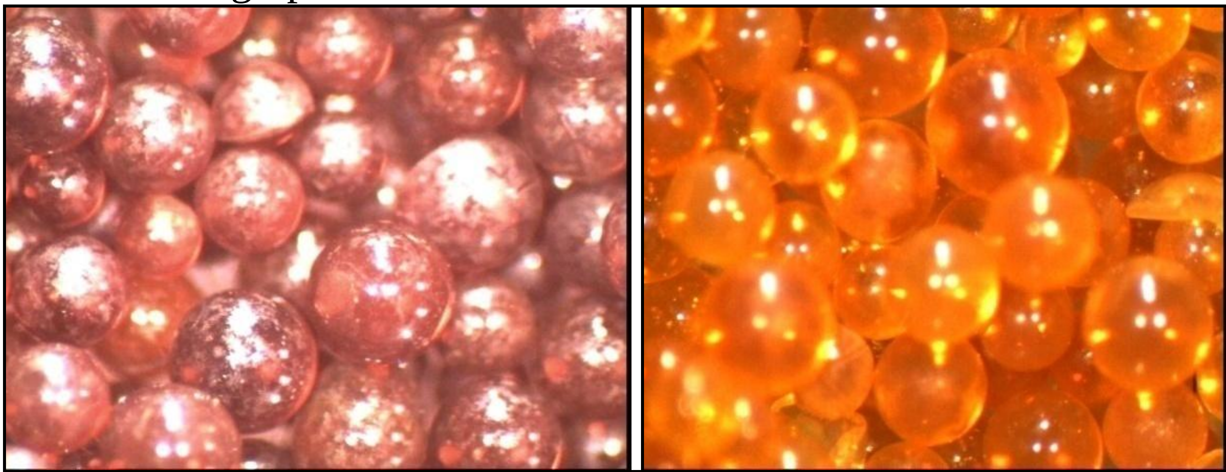
II-Materials and methods:

II.1-Sampling localities: Five samples were collected, in clean stopper bottles in August 2014, and used to pass through demineralization units. These were I- Raw Nile water. II-Water sample after clarification. III- water sample after sand filtration.

IV-Water sample after activated carbon filtration, before entering the demineralization plant. V-Fouled resin collected from the main anion vessel (Ten years in service). All the samples were delivered to lab within (6 hours) to ensure a meaningful report.

II.2.1-Fouled Risen:

The organic fouled anion resin sample was collected from anion vessel after ten year in service. Figure (1) shows the Photomicrograph of the fouled anion resin and new one.



Fouled anion resin	New anion resin

Figure (1): Photomicrograph of fouled and new anion resin.

The anion resin has the following specification:

Item	Specifications
Name	Strong Anion IRA-402
Matrix	Styrene divinylbenzen copolymer
Functional groups	$-N^+(CH_3)_3$
Physical form	Pale yellow translucent beads
Ionic form as shipped	Chloride
Total exchange capacity	≥1.30 eq/l(Cl^- form)
Moisture holding capacity	49 to 55 % (Cl^- form)
Specific gravity	1.063 to 1.093 (Cl^- form)
Shipping weight	670 g/L
Uniformity coefficient	≤ 1.6
Harmonic mean size	600- 750 µm
Fine contents	□ 0.300mm :1.0%max
Coarse beads	□1.180mm :5.0%max
Maximum reversible swelling	$Cl^- \rightarrow OH^-$: 30%
Maximum operating temperature	60°C
Regenerant	NaOH
Regenerant concentration	2 to 4%
Minimum contact time	30 minutes

II.2.2-Demineralization unit:

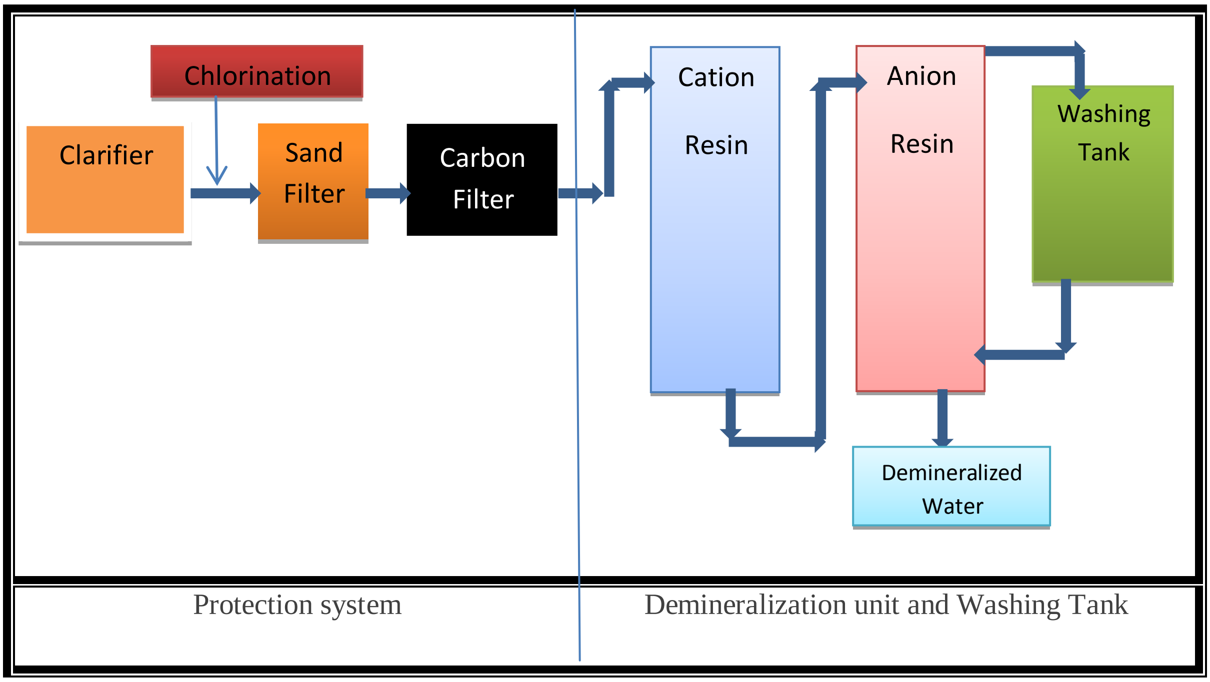


Figure (2) illustrate the schematic diagram of demineralization unit and protection system that used in this work

Figure (2): Demineralization unit and protection system.

II.2.3-Protective system:

Clarification by coagulation and filtration are the most effective systems to reduce the organic matter present in water (***Archer, A., Singer,P.C.,2006a and Archer ,A., Singer,P.C.,2006b***).Therefore the protective system in this work consists the following**:**

1. Clarification system
2. Filtration system.
3. Carbon filters system.

II.3-Analysis of inlet water (raw Nile water):

This test was carried out over a period of a year from January to December, 2014. Turbidity, electric conductivity (µS/ Cm^{-1}), colloidal silica (SiO_2), total solids (mg/l) and the most probable number of the coliform bacteria (MPN) were estimated according to the standard methods of ***ASTM (2001)***. The organic matter contents were evaluated as ($KMnO_4$) according to ***(Babcock 1982***).

II.4-Cleaning solutions (Brine solutions):

Eight cleaning solutions of NaCl, NaOH or NaOCl were used mixed or separately. These were:

(1)- 5% NaOH.

(2)- 5% NaCl.
(3)- Mixture of 5% NaOH and 5% NaCl .
(4)- Mixture of 5% NaOH and 10 % NaCl .
(5)- 1% NaOCl.
(6)- Mixture of 1% NaOCl and 5% NaOH.
(7)- Mixture of 1% NaOCl and 5% NaCl .
(8)-Mixture of 1% NaOCl , 5% NaOH and 5% NaCl.

II.5-Cleaning processes and technique:

The most common forms of brine treatment involve the use of brine solution; the procedure is as follows:-

1. The resin treated at the end of the normal exhaustion cycle.
2. One bed volume of the cleaning solution (Brine) introduced into the ion exchange unit at a flow rate not exceeding 2 BV's per hour followed by a second bed volume.
3. The second bed volume retained in the unit for as long as possible, but at least 4 hours or until the drained chemical colour changed from brown to very light yellow then the feeding of chemicals stopped.
4. At the end of the retention period the last bed volume of brine passed through the resin at a rate of 1 BV per hour and the resin thoroughly rinsed with demineralized water until free from brine.
5. The resin subject to at least two complete regeneration cycles before being put back on line.
6. Temperature of brine solution (cleaning solution) at minimum 35˚C employed or preferably as high as 45˚C so as to produce a better organic elution effect.

II.6- A preliminary cleaning technique of the fouled anion resin:

This experiment was carried out in laboratory. A sample of the fouled anion resin, taken from the main anion vessel, was introduced together with each of the cleaning solutions in stoppered flasks (in triplicates) and shaken for 1 hour. The percentage removal of organic matter from fouled resin was estimated after every brine treatment.

II.6.1-Cleaning technique of the fouled anion resin in the pilot demineralization plant:

a. Cleaning in the main anion vessel:

The fouled anion resin bed, present in the main vessel, was cleaned by continuous feeding of the most suitable cleaning solution, determined from the above preliminary cleaning test. The cleaning process continues until the drained chemical colour changed from brown to very light yellow then the feeding of chemicals stopped. The chemicals remain inside the vessel of anion resin for a period of 24 hour. Then the excess chemical will be rinsed using demineralized water; this rinse ends when the inlet water pH and colour are similar to the outlet. Thereafter the cleaned resin will be regenerated using 4% NaOH (double regeneration). After regeneration, the resin will be rinsed using demineralized water. The rinse time, the produced effluent conductivity and the number of service cycles (operation cycles between two cleaning processes) of the cleaned resin will be compared with that of both fouled and new resin of the same type.

b. Cleaning in the washing tank:

Washing tank is not present in the most demineralization plants. However, it is found in the pilot plant. In this investigation, we tried prewashing the fouled anion resin in the washing tank, before chemical cleaning. The fouled resin will be transferred, via resin transfer lines, to the washing tank and a current of demineralized water will be forced on the resin by feeding from top and bottom side, in a manner that makes a turbulent flow. After this prewash, the resin will be cleaned by the chemical solution. The effluent conductivity, rinse time and number of service cycles of the cleaned resin in presence or absence of washing tank was compared with that of the new resin of the same type.

II.7-Iron/Organic matter Complexes cleaning:

This subject is covered to some extent in the section on iron fouling.Occasionally the presence of iron is detected on the anion resin. This can arise from an iron/organic matter complex being present in the raw feed water.In these cases; it is advisable to consider treatment of the anion resin with 4% hydrochloric acid immediately after the brine treatment. The procedure that should be followed is similar to that given for brining.It is extremely important that all traces of hydrochloric acid are removed from the unit before indroduction of the caustic soda regenerant.

III-RESULTS AND DISCUSSION:

III.1-Physicochemical analysis of inlet raw Nile water:

The analyses of water source (Raw, clarified, after sand filter and after carbon filter) are listed in table (1) in the average reading over one year.

Sample / Analysis	Raw water	Clarified water	Filtered water	Carbon filtered water
PH	8.4	7.7	7.8	7.8
Conductivity(µ.s.cm^{-1})	296	315	316	316
Total hardens (mg/l) as $CaCO_3$	110	106	106	106
Calcium hardens (mg/l)	70	71	72	72
Magnesium hardens (mg/l)	40	35	34	34
Cl^- (mg/l)	13.4	13.3	13.5	13
SO_4^{-2} (mg/l)	21	30	31	29
Organic matter as $KmnO_4$	10.9	8.5	6.9	3.9
SiO_2^{-2}	5.2	4.6	4.54	4.5
Fe^{+2}	0.025	0.022	0.02	0.02
T .D.S (mg/l)	201	201	202	202
Turbidity NTU	4.7	1.5	0.97	0.7
Na^+ ($CaCO_3$)	48	48	50	50
K^+ ($CaCO_3$)	6	6	6	6

It is well known that anion resins are susceptible to fouling by the humic and fulvic acids sometimes found in surface waters. These organic species, because of its charge and the relatively large molecular weights, become trapped within the resin matrix to a greater or lesser degree depending upon the resin***(Kim, P.H.S., and Symons, J.M., 1991. and Lee, N.,et al., 2003)*** and specific procedures have to be employed to cause recovery of the original ion exchange properties of the resin.

The symptoms of organic fouling include long rinse time requirements, poor capacity and, in the case of strong base resins, higher silica leakage.

When comparing the analysis of raw water during one year of test. The results revealed that neither the pH (Fig.3) nor the conductivity (Fig.4) of the raw Nile water changed all over the year of test, from January to December 2014. This observation indicates that the inorganic compounds (salts) were considerably constant during the operation time. However, the organic matter content was higher in summer months, particularly August than in winter (January). This was also the case in turbidity, colloidal silica (Fig.3), most probable number of coliform (MPN) and total solids (Fig.4).

Obviously, these values are related to each other and the most effective value is the organic matter i.e. the increase in its value leads to the increase in the others. Nile water contains a variety of contaminants including organic matter derived from biological (***Boyer, T.H., Singer, P.C., 2008 Escobar, I.C.et al., 2001***), industrial pollutants ***(Aivasidis et al., 1992),*** oily wastes ***(Mercer, K., et al., 2004)*** and others.

Interestingly, during the period of test, the increase in the demineralization plant operation problems, in power station, is correlated with the increase in the quantity of organic matter in summer months. This observation indicates and confirms the principal role of microorganisms, which greatly multiply and reproduce at this season, in exerting organic fouling of anion resin, in addition to the other organic matter ***(Betz, 1991)***.

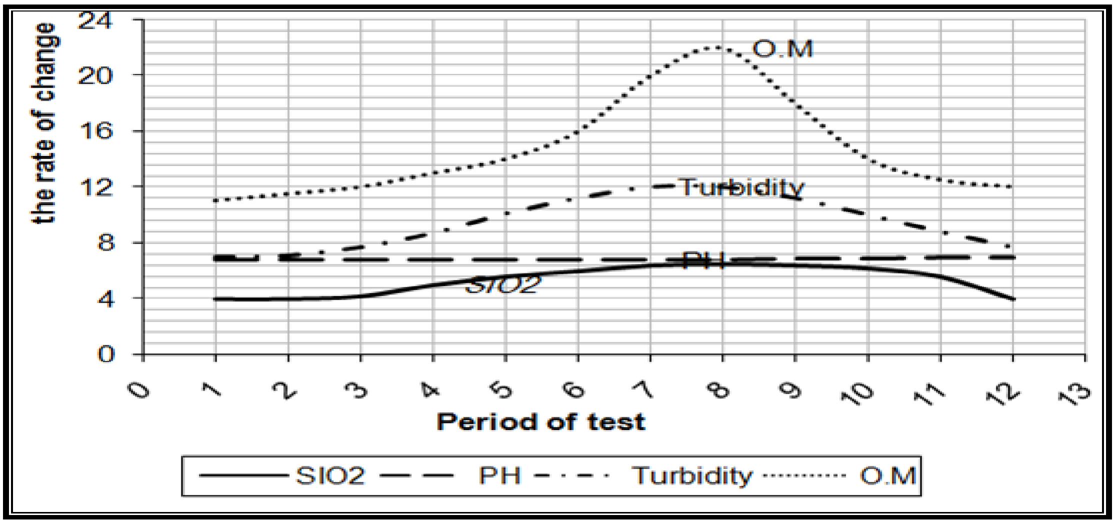


Figure (3): Variation of SiO_2 mg/l, pH, Turbidity (NTU) and organic matter mg/l during one year from January (1) to December (12) 2014.

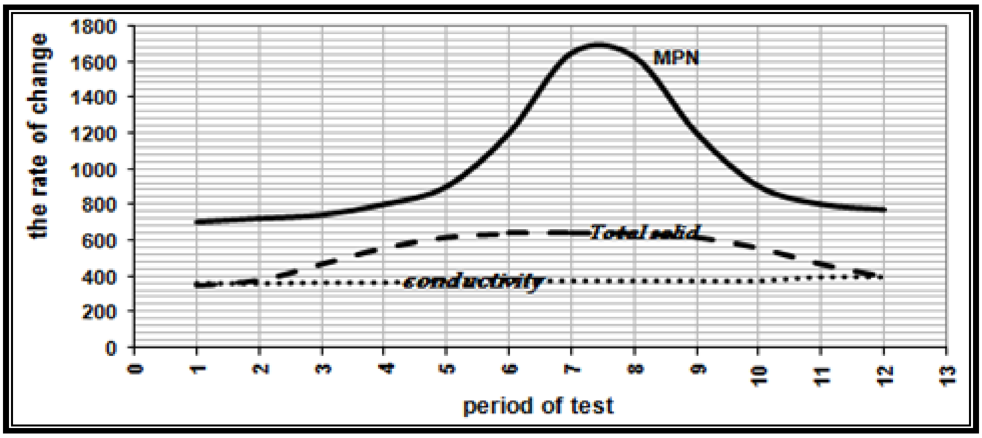


Figure (4): Variation of Conductivity µ.s.cm^{-1}, Most Probable Number (MPN) and total solid mg/l of Nile Water during one year from January (1) to December (12) 2014.

III.2-Cleaning technique of fouled anion resin:

The removal efficiency (% Removal) was calculated from the following formula:

$$\% \text{ Removal} = C_o - C / C_o \times 100$$

Where, (C_o) is the initial value and (C) is the final value of organic matter and iron contents on fouled resin before and after cleaning. The results of cleaning a sample of fouled resin in laboratory revealed that all the cleaning solutions could remove organic matter and iron but to a variable degree. Figure (5) shows the efficiency of cleaning solution on the removal of organic matter.

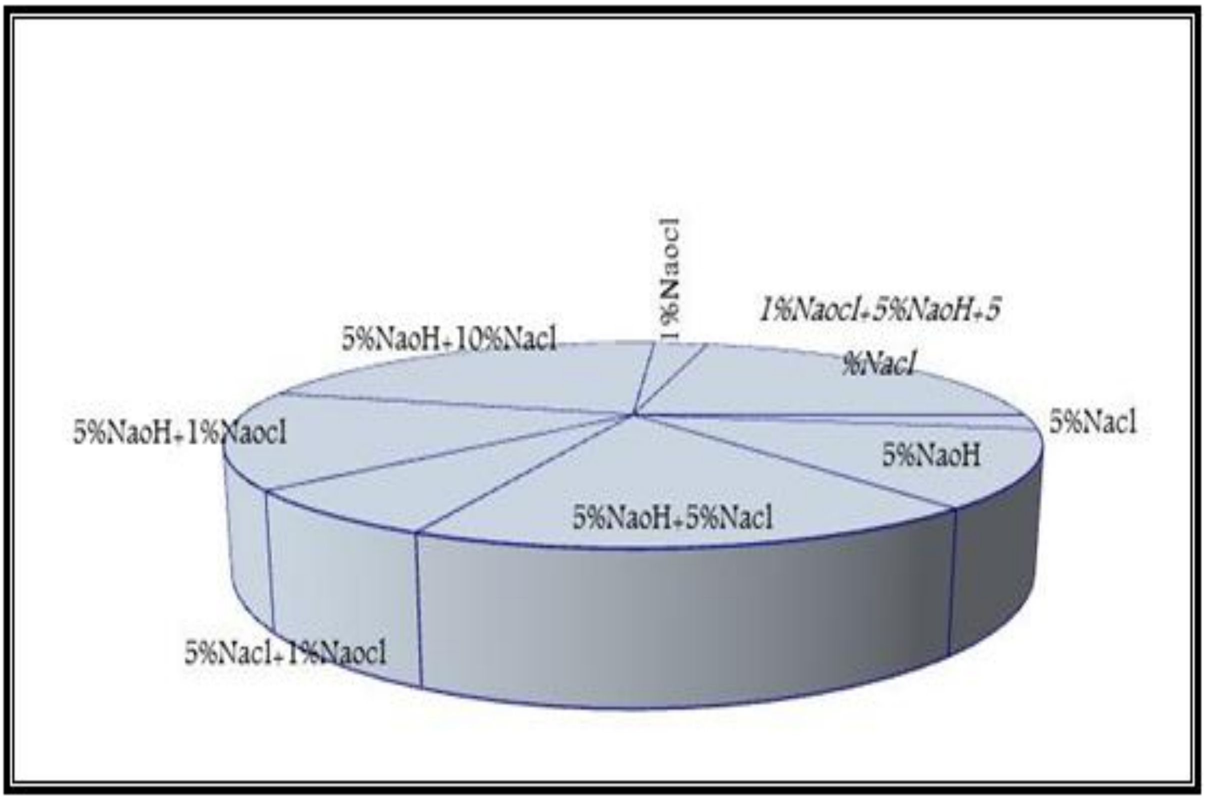


Figure (5): Effect of different cleaning solutions on the removal of Organic Matter And iron from a sample of the fouled anion resin.

However, the maximum removal capacity of organic matter was achieved by a mixture of 5% NaOH and 10% NaCl (about 98%) and a mixture of 1% NaOCl and 5 % NaOH and 5 % NaCl (about 96%).

It is worthy to mention that, traditional separate cleaning solutions of NaOH, NaCl or NaOCl might leave some fouling on the resin particles so they could degrade and deteriorate it, while a mixture of 5% NaOH and 10% NaCl might exert a dual action i.e. not only remove the organic matter comes from microorganism but also kills them, thus it could clean and protect the resin from deterioration.

III.3-Effect of cleaning solutions on the specification of cleaned resin:

The effects of cleaning solutions (Brine treatment) on the specifications of the anion resin are shown in table (3). It is clearly seen from table (3) that all the parameters of resin were improved and become equal to the new resin especially for the cleaning solution number 7, 8. Also the physical properties of all resins after cleaning by the different cleaning solution were enhanced.

Table (3):Effect of cleaning solutions on the specification of cleaned resin.

Item	Specifications of New resin	Specifications of fouled resin	Specification of cleaned resin*							
			1	2	3	4	5	6	7	8
Total exchange capacity eq/l	≥1.30	1.1	1.11	1.13	1.2	1.22	1.25	1.25	1.35	1.4
Moisture %	49 to 55	57	55	55	54	54	54	54	54	54
Specific gravity	1.063 to 1.093	1.063 to 1.093	1.1	1.1	1.1	1.1	1.09	1.09	1.09	1.09
Shipping weight g/L	670	660	660	660	665	665	660	660	660	660
Uniformity coefficient	≤ 1.6	≤ 1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Harmonic mean size µm	600- 750	600- 750	700	700	700	700	700	700	700	700
Fine contents (□ 0.300mm)	1.0 % max	1.0 % max	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
Coarse beads(□1.180mm)	5.0%max□	5.0%max□	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Maximum reversible swelling $Cl^- \rightarrow OH^-$	30%	30%	20	20	20	20	20	20	20	20

***CleaningSolution:1-(5%NaCl)-2-(1%NaOCl)-3-(5%NaCl+1%NaOCl)-4-(5%NaOH)-5-(5%NaOH+1%NaOCl)-6-(5%NaOH+5%NaCl)-7-(1%NaOCl+5%NaOH+5%NaCl)-8-5%NaOH+10%NaCl).**

III.4-Effect of protective methods and cleaning:

Flocculation –filtration:

Flocculation provides efficient protection against organic matters. Indeed the organic matters removed by flocculation are often that which are harmful to the ion exchange resins. Also sand filter can reduce the load of organic matter and gives water with low organic matter contents.

Activated carbon:

Activated carbon is effective, particularly when the water has been pre-chlorinated, and can remove the most harmful organic matters .its fixation capacity is good.

III.4.1-Check the efficiency of the protective system:

The protective system is generally accepted as being effective when the percentage of fixation of organic matters is high. In calculating this percentage the organic matters content has to be measured usually by the permanganate method.

The degree of harm on anion resin caused by organic matters depends on its nature; for instance, sugars and alcohols increase the quantity of organic matters measured by the permanganate method, but do not become fixed on ion exchange resins.

In this case, therefore, the percentage of fixation may be low, but will not thereby cause fouling of the strongly basic exchange resin.

The method of measuring humic acids by absorption of ultra-violet rays is certainly more specific, and therefore to be preferred to the permanganate method; but other products both ionic and non- ionic can be absorbed at the same wave-length and it is impossible to be sure that a poor fixation result necessarily means that the strong anion exchanger will become fouled.

The only way to judge the real efficiency of the protection is by the performance of the anion exchange resins; if the quality of the water produced is good, if the anion exchange use reasonable quantities of rinsing water without becoming sensitive to an increase in flow- rate and if the strong anion exchanger does not rapidly lose its useful capacity, it may be concluded that the protection afforded is effective.

Figure (6) illustrates the results obtained with water containing (10.9 mg/l) of organic matters ($KMnO_4$) and (0.025mg/l iron) in the absence and presence of protective system.

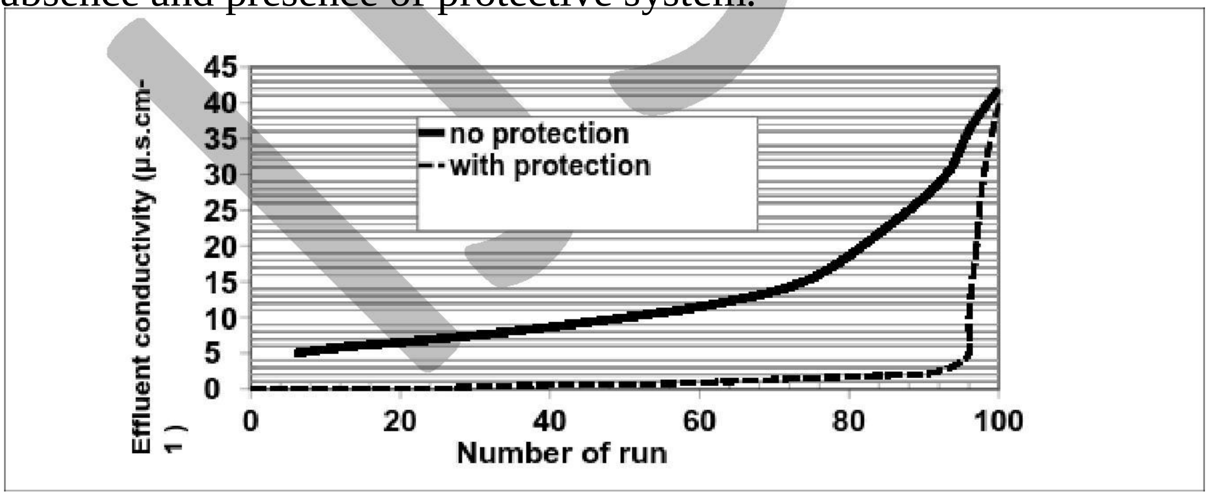
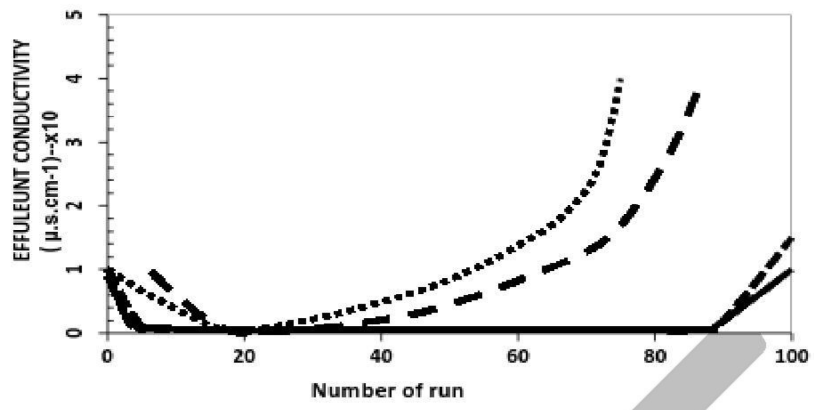


Figure (6): No. of run against variation of conductivity of resin cycle.

The unit with new anion resin operates at least 90 run until cleaning should be done in case of the presence of protective system. As can be seen from figure(7) in case of no protection the unit gives water with poor quality and the number of run reduced to about 50 run at which the effluent conductivity was 10 (μ.s.cm^{-1}).



….. Cleaned anion resin with no protection.
- - - - New anion resin with no protection.
— — —Cleaned anion resin with protection.
——.noitcetorp htiw niser noina weN -

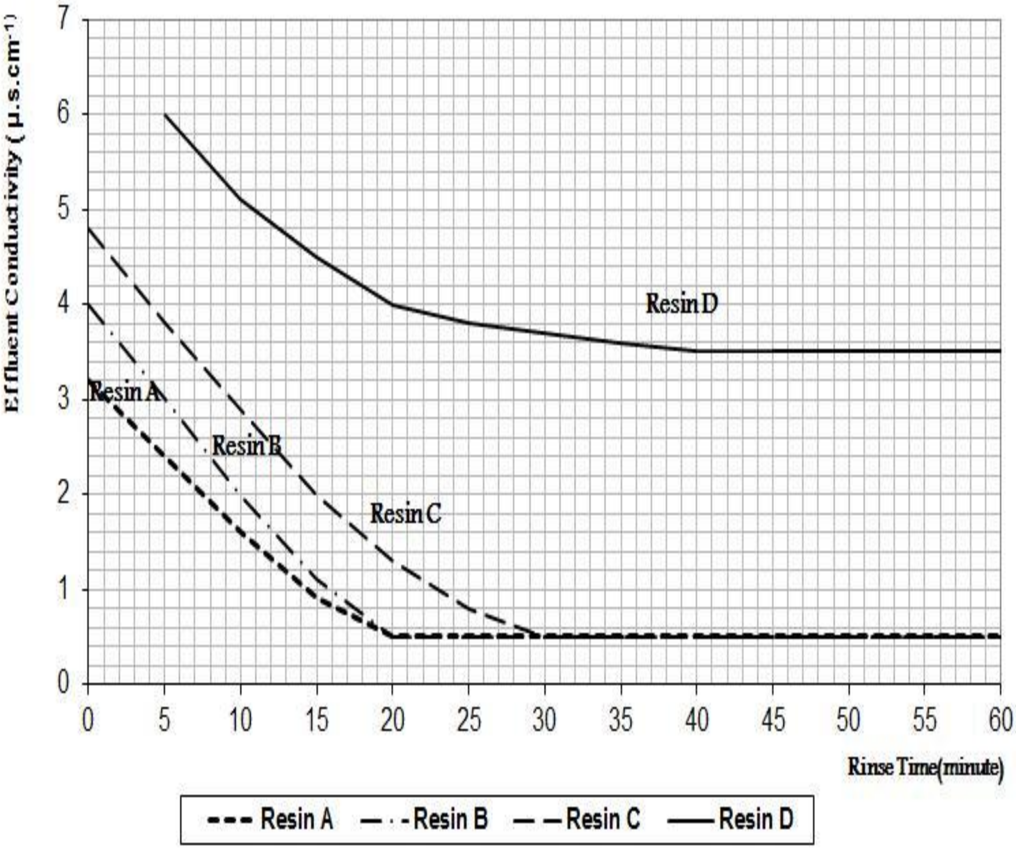
Figure (7) Comparison between the No. of run between tow cleaning processes with and without protective system**.**

III.4.2-Cleaning technique in the pilot demineralization plant:

Virtually, a mixture of 5% NaOH and 10 NaCl was more preferred than a mixture of 1% NaOCl and 5% NaOH and 5% NaCl for the following advantages: the used chemicals are costless, don't need any especial device, don't deteriorate the resin and they are available at any time. Moreover NaOCl, present in the unpreferable chemical, despite it could destroy the microorganisms and organic matter, it also significantly degrades the resin, and therefore it is considered a last resort (***Betz, 1991)*.**

Therefore, the best cleaning solution 5% NaOH and 10 % NaCl, which achieved the maximum removal of organic matter, microorganisms included, was used to clean the fouled anion resin. The prewash of the resin in the washing tank, before the chemical cleaning, achieved a better cleaning technique than if applied in the main anion vessel. The long rinse time and high effluent conductivity are the permanent feature of the service cycle suffers from resin fouling. The rinse time (20 mints) and effluent conductivity (0.3µ scm^{-1}) of the cleaned resin, which is prewashed in the washing tank, were similar to that of the new resin of the same type. The effluent conductivity of the cleaned resin, applied in the main vessel, was also the same as that of the new resin, but after a long rinse time (30 mints or more).

The reduction in the rinse time of the prewashed resin, in the washing tank, is noteworthy. The turbulent flow of the demineralized water, feeded to the washing tank, helps the separation of the aggregated fouled resin particles, so they become widely spaced and easily exposed to the cleaning solution, a technique that enhances and facilitates the chemical cleaning process (Fig 8).



Figure(8):Rinse time of service cycles of new resin (A), resin cleaned by using washing tank(B), resin cleaned in the main vessel without washing tank(C) and fouled resin (D).

After 90 cycles, the rinse time begins to increase, a monitor for resin fouling. Prewash and cleaning the fouled resin in the washing tank achieved results very near to the new resin i.e. 90 service cycles, with rinse time 20 mints. However, when the cleaning process was applied in the main vessel, the number of service cycles were only 70 cycles, with rinse time 30 minutes or more. These observations emphasized the role of washing tank in improving the cleaning process of the fouled resin.

Concerning the number of service cycles, the new and cleaned resin by solution number (8) gives 90 cycles that still have 20 minutes rinse time (Fig.9).

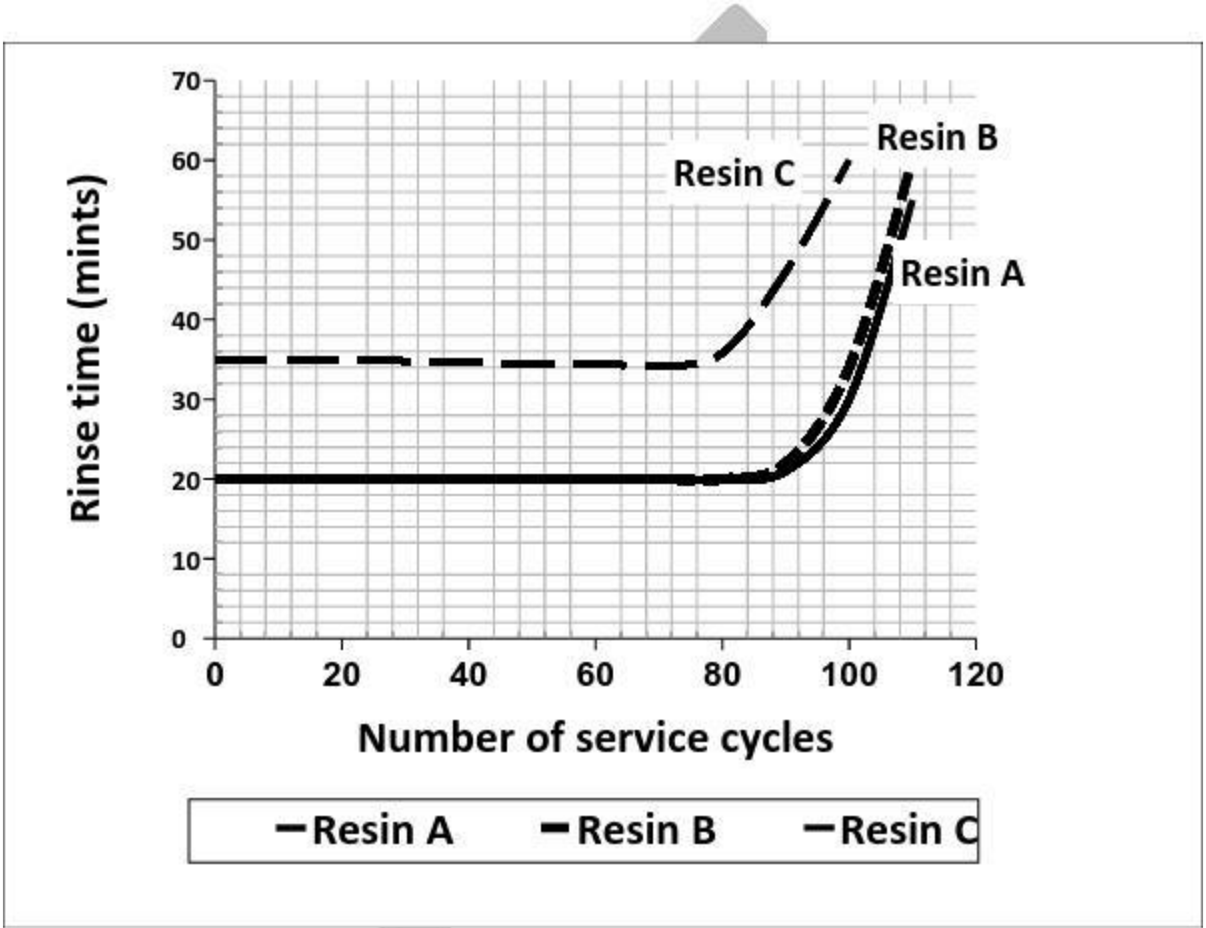
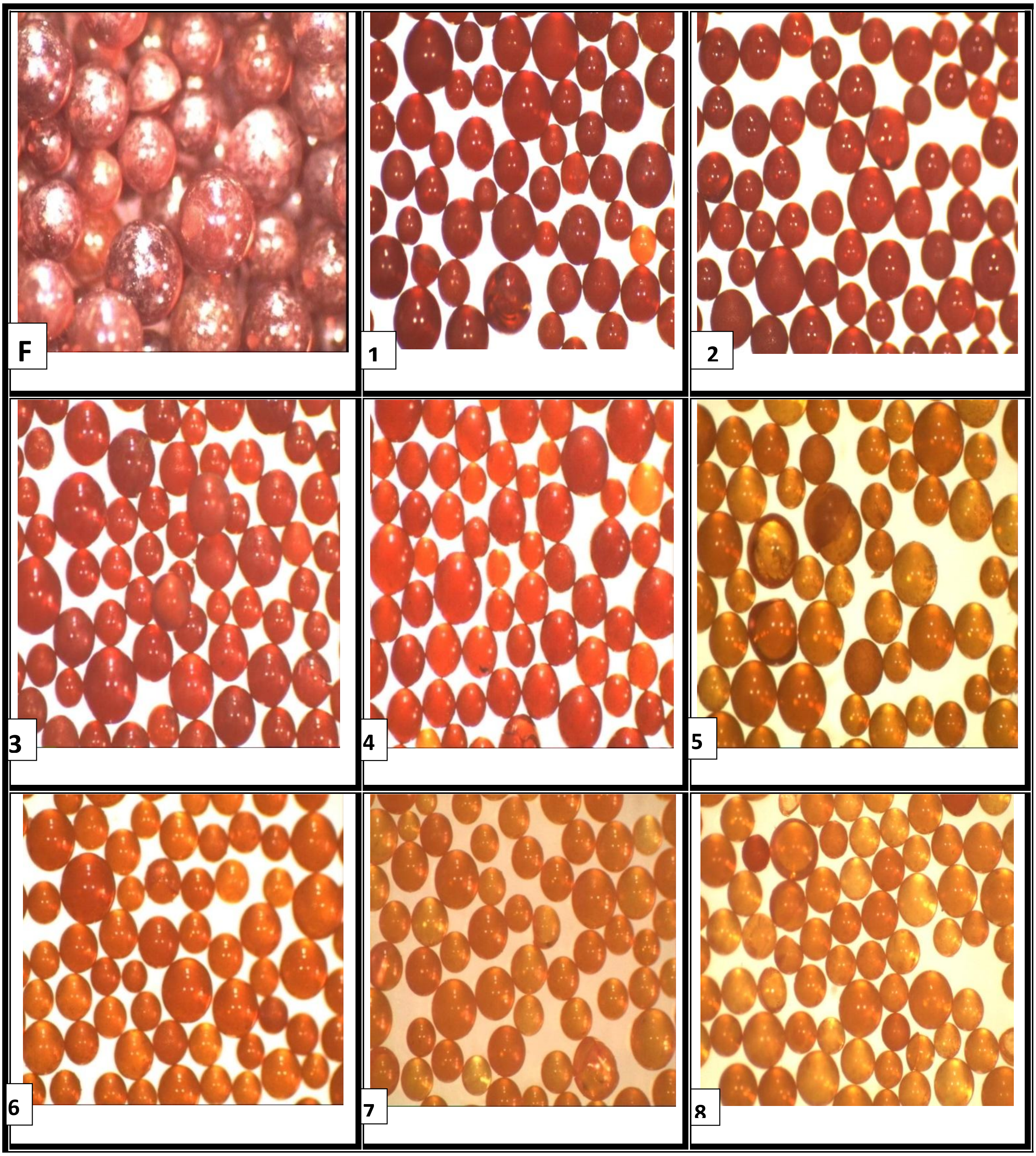


Figure (9): Variation of number of service cycle (no. of runs) and rinse time of new anion resin (A), cleaned anion resin (B) and fouled anion resin.

III.5-Photomicrographs of fouled and cleaned resin using different kinds of cleaning solutions:

The work was extended to show visually the efficiency of cleaning solution (Brine treatments) using different types of cleaning solutions.



***CleaningSolution:1-(5%NaCl)-2-(1%NaOCl)-3-(5%NaCl+1%NaOCl)-4-(5%NaOH)-5-(5%NaOH+1%NaOCl)-6-(5%NaOH+5%NaCl)-7-(1%NaOCl+5%NaOH+5%NaCl)-8-5%NaOH+10%NaCl).**

Figure (10): Photomicrograph of fouled and cleaned anion resin using different kind of cleaning solutions.

IV- Conclusion:

Ion exchange resins can become polluted or contaminated over time with suspended solids, slime, bacteria, or numerous other kinds of organic or microbiological matter. Adsorption or ion exchange of other species which are not easily removed by the normal regeneration procedures can cause gradual performance losses due to accumulation and fouling of the resin sites. Typically, organic matter and iron fouling of the ion exchange resin is the most common type of fouling.

From the above results and discussion, it could be suggested that the process of raw water pre-treatment, before entering the demineralization plant, should be increased to remove the most part of organic matter and microbes that are the main source of resin fouling. Also, the use of the cleaning mixture of 5% NaOH and 10 % NaCl could rejuvenate and protect the anion resin from deterioration by these contaminants, and this process will be more satisfactory if the resin is prewashed in a washing tank.

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