# Article

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Development of an Evaluation Technique Inhibitors of NaCl Salt Deposit Encountered in Oil Production

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#### Abstract

Oil production is one of the activities that potentially cause pollution and environmental damages, the largest waste generated from this activity is water used in washing of the salted wells in order to fight against the NaCl solid deposits, salts deposits are a major problem encountered in producing oil. In general, the salts deposits will cause a reduction in formation pores, declining productivity and eventually blockage of the wellbore and hence unexpected downtime if it is allowed to persevere. The water used for washing is drawn from fresh groundwater. These waters once used are thrown back into nature with their polluting hydrocarbons load, killing the life of water and plants. In order to the objective to reduce the volume of waters used in the washing of the salty wells, we tested in the laboratory three methods to estimate the efficiency of the inhibitory products of the NaCl deposits. The obtained results show that it is possible to select in the laboratory the appropriate treatment products. The use of these products will allow us to reduce the volumes of water used for the washing of salts. This reduction is synonymous of a reduction of the pollution on one hand, and a protection of our water resources on the other hand.

Keywords: Deposit inhibitor; NaCl Deposit; Production of oil; Water for washing; Pollution of environment.

#### 1. Introduction

Salt deposits are a major problem encountered in producing oil <sup>[1]</sup>. In general, the salt deposits will cause a reduction in formation pores, declining productivity and eventually blockage of the wellbore and hence unexpected downtime if it is allowed to persevere <sup>[2-3]</sup>.

The salt deposit formation process has been reported to involve four phases <sup>[4]</sup>: aggregation, nucleation, crystal growth, and agglomeration. Aggregation occurs after the brine system is supersaturated with scaling ions, cationic and anionic species collide to form ion pairs in solution and subsequently form micro-aggregates. The micro-aggregates develop into nucleation centers for crystallization which lead to the formation of micro-crystals in the nucleation stage. These micro-crystals develop into larger micro-crystals and eventually fuse to form adherent macro-crystals as they agglomerate and/or absorb to surfaces in crystal growth stage. The agglomeration stage is marked with the growing of macro-crystals into a salt deposits <sup>[5]</sup>.

Producing wells were confronted by a severe salt deposits problem, more or less related to the exploitation regime, which severely reduced oil production. The formation of these salt deposits poses several serious threats in oil operations. They can cause technical problems such as the clogging of equipment and pipes, which causes great damage and leads to economic losses <sup>[6]</sup>.

The formation waters in oil fields are supersaturated with salts <sup>[7]</sup>. This water, as it rises in the tubing during production, deposits salts on the inside wall of the latter until the tubing and also the surface installations are completely blocked <sup>[8-9]</sup>. We cite the cases of the Field of Bir R'Baâ Nord (BRN), the Field of Hassi Messaoud and that of Oued Noumer (Region of Hassi-R'Mel) from south of Algeria, where the producer is constantly confronted with the problem of solid deposits of NaCl.

The presence of sodium chloride inorganic salt deposits in the well is undoubtedly influenced by the type and quantity of water influx; operating procedure; and reservoir characteristics, such as temperature and pressure <sup>[10]</sup>.

A permanent fresh water injection under a packer did not improve the performance of these wells <sup>[11]</sup>. A large volume of water injection is required for particular wells, tubing intervention. These injection waters contain hazardous pollutants of both organic and inorganic materials, are discharged directly to the environment <sup>[12-13]</sup>, killing the life of water and plants <sup>[14]</sup>.

Relatively low concentrations of water-soluble inorganic and organic compounds are known to reduce the rate of depot formation in producing wells in the petroleum industry <sup>[15]</sup>. A chemical compound that prevents or retards mineral depot from forming in brines saturated with a pair of scaling ions by chelating scaling cations and/or modifying the growing scale crystal is referred as a "inhibitor" <sup>[16]</sup>. A salt deposit inhibitor can simply be defined as a chemical substance which, when added in small amounts into the brine system, reduces or prevents depot from forming <sup>[17]</sup>.

Application of scale inhibitors is proven to be an economic and cost-effective approach for combatting scale deposition in many fields. Until now, many chemicals naturally occurring <sup>[18]</sup>, semisynthetic <sup>[19]</sup> or synthetic <sup>[20]</sup> have been proposed and successfully tested in the laboratories and/or in the field as scale inhibitors, and many of them have even been patented <sup>[21]</sup>.



Figure 1. NaCl salt deposit

with the, laboratory division (DL) at Sonatrach to solve the salt deposit problem in production due to the effect of temperature and pressure change. It consists of analyzing a sample of the formation water in order to understand the phenomenon of the salt deposit, to test the anti-deposition product efficiency, and to optimize its concentration under surface and bottom conditions, keeping in mind the reduction of the amount of injection water. Laboratory testing was undertaken first of all, in order to develop a technique to evaluate the effectiveness of products that inhibit NaCl salt deposits, before testing them on site. Figure 1 shows the NaCl salt deposits.

A study was undertaken in collaboration

## 2. Experimental part

## 2.1. Materials

All the material and reagents used were provided by the water treatment service of the Laboratory Division of Sonatrach. All the experiments were carried out in the water treatment laboratory of the laboratory division of the Sonatrach group. An assembly carried out in the laboratory then enabled us to proceed to semi-pilot tests, to verify (confirm or refute) our hypotheses made on the basis of the first experimental results.

## 2.2. Methods for evaluating NaCl salt deposit inhibitors

Salt deposits inhibitors are customarily evaluated in the laboratory prior to deployment into an actual field. The evaluation is aimed at choosing a suitable inhibitor candidate, which will effectively and economically prevent/retard scaling for an actual field. Currently, there is no general method, standardized or not, to assess the effectiveness of products that inhibit deposit formation by "Self-Scaling". A method of proven reliability has been developed in the Laboratory Division of the Sonatrach Group, to evaluate the efficacy of inhibitors of calcium sulphate deposits (CaSO<sub>4</sub>,  $nH_2O$ ), but no method has been tested or developed to evaluate the effectiveness of inhibitors of sodium chloride (NaCl) deposits. Currently, salt deposit inhibitors are used at BRN to wash some wells, but the treatment concentrations are recommended by the supplier.

We tested three methods in this work: the first is the one proposed by a supplier of treatment products, the other two were proposed by the Products and Treatment Laboratory of the Laboratory Division.

The three methods are based on the same principle, that of preparing supersaturated NaCl solutions, under defined temperature and pressure conditions, adding the deposit inhibitor product at the desired treatment concentration and then observing and after cooling at room temperature and at atmospheric pressure, the morphology of the NaCl crystals formed, with an evaluation of the mass of the crystals formed. The results obtained and the observations made are compared with the results and observations recorded on a control test.

Sodium chloride naturally crystallizes in the symmetrical cubic system. This form of crystallization leads to the formation of large crystals which agglomerate in a compact and translucent mass.

In practice and in oil production, the agglomeration of crystals often forms plugs that obstruct production lines. A salt deposit inhibitor changes the crystallization system from cubic to dendritic. This latter crystallization system is characterized by the formation of very fine crystals unable to agglomerate and which are carried away by water currents as suspended matter, without risk of clogging.

#### 2.2.1. First method: proposed by the supplier

The principle of the method is based on the action of the product to be tested on the morphology of the salt crystals. After hot dissolving 55 g of NaCl in 145 ml of water, the solution is cooled to room temperature while monitoring the mass and shape of the supersaturated salt crystals formed.

Put 55 g of sodium chloride (NaCl) previously dried at 105°C for at least two hours in a 250 mL conical flask, add 145 mL of water. Place the flask on a magnetic stirrer and heat with continuous stirring until boiling. Stop stirring and heating, close the conical flask and allow it to cool to room temperature. The solution prepared is a supersaturated solution containing approximately 328 g NaCl/L of solution or 380g NaCl/L of water. Repeat the same test, but adding an amount of NaCl deposit inhibitor to the solution for a treatment concentration of 100 ppm.

The solution is then transferred to a graduated separating funnel with a conical bottom to evaluate the quantity of NaCl crystals formed as a function of time. The crystals formed are then examined with the naked eye and under an optical microscope to define their morphology.

## 2.2.2. Second method: proposed by the Laboratory Division of the Sonatrach group

A volume of liter of aqueous solution supersaturated with 380 g/L of NaCl is prepared under the same conditions as those described in the first method. 100 mL volumes of this solution are then taken into 250 mL flasks. A precise volume of the NaCl deposit inhibitor product is added to each vial for the desired treatment concentration. The vials are sealed and placed at 80°C for 72 hours.

After cooling to room temperature, the salt crystals formed are subjected to the same observations and evaluations as those of the previous method.

## 2.2.3. Third method: proposed by the Laboratory Division of the Sonatrach group

This procedure is the same as that described in the second method, but the flasks with the supersaturated NaCl solution and the salt deposit inhibitor are placed under a pressure of 2 bar and at 120°C for 120 min. After cooling to room temperature, the crystals formed undergo

the same treatment as for two previous methods. Three inhibitors were tested: EC6151A, GYPTRON SA 2070 and AVA inhibitor.

## 3. Results and discussion

## **3.1. Results of the first method for the evaluation of deposit inhibitors**

The tests were carried out under the same conditions for the three inhibitor products tested. The experimental results are given in Tables 1, 2 and 3, where we present the chemical compositions of the test solutions, the masses of NaCl crystals formed and a description of their morphology. Observing the crystals under an optical microscope has allowed us to better describe their external morphology and to define their structure. The results for the three inhibitors are shown in Tables 1. 2, and 3.

Test solution + observations	Witness test	Test in the presence of the EC6151A inhibitor	
Water volume in mL	145	145	
NaCl mass in g	55	55	
NaCl concentration in g/L	380	380	
Deposit inhibitor volume in µL	0	14.5	
Treatment concentration in ppm V/V	0	100	
Mass of salt crystals formed in g	4.886	0.748	
Reduction of crystal formation in %	-	84.69	
Crystal shape and observations	Cubic and agglomerated crystals	Dendritic shaped crystals, thin and separated from each other	

Table 1. Results obtained with a solution supersaturated with NaCl treated with the product EC6151A

Table 2. Results obtained with a solution supersaturated with NaCl treated with the product GYPTRON SA 2070

Test solution + observations	Witness test	Test in the presence of the GYPTRON SA 2070 inhibitor	
Water volume in mL	145	145	
NaCl mass in g	55	55	
NaCl concentration in g/L	380	380	
Deposit inhibitor volume in µL	0	14.5	
Treatment concentration in ppm V/V	0	100	
Mass of salt crystals formed in g	4.886	0.748	
Reduction of crystal formation in %	- 84.69		
Crystal shape and observations	Cubic and agglomerated crystals	Cubic and agglomerated crys- tals	

Test solution + observations	Witness test	Test in the presence of the AVA inhibitor	
Water volume in mL	145	145	
NaCl mass in g	55	55	
NaCl concentration in g/L	380	380	
Deposit inhibitor volume in µL	0	14.5	
Treatment concentration in ppm V/V	0	100	
Mass of salt crystals formed in g	4.886	0.498	
Reduction of crystal formation in %	-	89.80	
Crystal shape and observations	Cubic and agglomerated crystals	No visible crystals	

Table 3. Results obtained with a solution supersaturated with NaCl treated with the product AVA

The results obtained differ from one product to another. Indeed, this test allowed us to identify three forms of action of treatment products and to make the following observations:

If the product is ineffective, the shape and quantity of the NaCl crystals remain essentially the same for the test as for the blank. This is the case with the product GYPTRON SA 2070. In this case, the product has no effect on the formation and on the interactions between the crystals of NaCl.

If the crystallization form and the mass structure of the crystals formed in the presence of the inhibitor are different from those of white, the product can be effective, as long as the mass structure of the crystals formed remains loose, and not under form of clods. This is the case with product EC6151A, for which we have observed a considerable reduction in the mass of the crystals formed. The crystals formed are very small, dendritic in shape and are separated from each other.

For the AVA product, we observed an almost total absence of the crystals which settle out of the supersaturated solution. The solution remains cloudy after cooling. This product therefore prevents the formation of crystals large enough to be able to settle quickly. In light of these observations, the effectiveness of this product seems obvious to us.



Figure 2. X-ray diffractogram of the solid deposit formed in the solution treated with the AVA product

The low mass of the solid deposit formed in the solution treated with the AVA product consists essentially of impurities provided by the sodium chloride. The deposit is dark and has a powdery texture. X-ray diffraction revealed an amorphous structure with a halite (NaCl) peak embedded in the dominant amorphous phase. The results are shown in Figure 2.

If for the last two cases we have observed a strong reduction in the formation of NaCl crystals, this absence or

reduction in the quantity of crystals is only temporary. In fact, when the supersaturated solutions are left to stand, the quantity and shape of the crystals change as a function of time.

#### 3.2. Evolution of the quantity and shape of crystals formed as a function of time

We carried out this test for a supersaturated solution of NaCl treated with the product AVA. The solution is prepared as in the previous procedure, then it is transferred into a graduated funnel with a conical bottom and left to settle in order to follow the formation of the crystals over time. The results are presented in Table 4 and Figure 3.



Figure 3. Photo showing the germination streaks on the crystals formed in the solution treated with the AVA product after 3 hours of rest

The results obtained show that the inhibitory action of the product on the formation of settled crystals is only temporary. The tiny crystals which were initially in suspension undergo a phenomenon of germination and end up settling while continuing to increase in volume.

Another parameter must therefore be taken into account when talking about the effectiveness of products that inhibit salt deposits, it is the duration of action of the product on the formation of salt crystals. Salt deposition inhibitors therefore have a limited action over time, similar to that of barium sulfate inhibitors.

Decantation time in hours	Volume of crystals formed in mL	Observations
0	0,00	Cloudy and stable suspension (no deposit formation)
0,5	0,00	No deposit formation
1	0,05	Precipitation of a few fine crystals of NaCl
2	0,80	Appearance of sticks
3	1,02	Presence of small cubic crystals
5	2,00	Large rod crystals predominate
24	3,00	Large rod crystals predominate
48	3,00	Stabilization of the amount of crystals formed

Table 4. Monitoring of the amount of crystals formed as a function of time

#### **3.3. Results of the second method for the evaluation of deposit inhibitors**

After filtration of each solution on a pre-weighed filter, the mass of the NaCl crystals is determined by differential weighing, then the crystals are observed under an optical microscope. The results are given in Table 5. Figures 4, 5 and 6 show NaCl crystals formed in a supersaturated solution treated with the three inhibitors.

Table 5. Mass of salt crystals formed in the absence and presence of inhibitors (T= $80^{\circ}$ C, atmospheric pressure)

Inhibitor concentration in ppm	EC6151A	SA 2070	AVA
	Mass of salt crystals in g		
0	3.880	3.880	3.880
1	3.143	3.290	3.066
2	2.987	3.259	2.945
3	2.837	3.465	3.455
5	2.785	3.269	2.458
10	2.720	3.155	3.168

The amount of crystals formed, their structures and their cohesion to each other are the parameters that reflect the degree of effectiveness of a product.

Examining these parameters makes it possible to judge the effectiveness of a product and also to compare the products tested with each other from an effectiveness point of view.



Figure 4. NaCl crystals formed in a supersaturated solution treated with the product EC6151A at 50 ppm



Figure 5. NaCl crystals formed in a supersaturated solution treated with the product SA 2070 at 50 ppm



solution treated with the product AVA at 50 ppm



Figure 6. NaCl crystals formed in a supersaturated Figure 7. NaCl crystals formed in a supersaturated solution treated with the product EC6151A at 50 ppm

For an effective product, the amount of crystals formed is significantly lower in the treated solution than in the control test, so the crystals are separated from each other (loose structure), they are small in size with dendritic shapes (Table 5, Figure 7).

The less effective the product, the greater the guantity of crystals formed and the more the crystals are assembled into lumps of more or less solid cohesion. The crystals formed in the solution treated with the product SA 2070 are agglomerated into lumps which are difficult to crumble by simple pressure of the fingers.

Unlike the previous method, where the solution treated with the AVA product did not show any crystals visible to the naked eye, we obtained with this method a quantifiable mass of crystals. This would certainly be due to the time-limited action of the treatment product.

## 3.4. Results of the third method for the evaluation of deposit inhibitors

The results relating to this method are given in Table 6. Figures 7, 8 and 9 show NaCl crystals formed in a supersaturated solution treated with the three inhibitors. The results obtained showed the same observations as those obtained in the second method. However, we have noticed that the amounts of salt crystals formed are greater. The relatively long cooling period in this case would be the cause of the formation of larger amounts of crystals.

Inhibitor concentra- tion in ppm	EC6151A	SA 2070	AVA
	Mass of salt crystals in g		
0	3.880	3.378	3.378
1	3.143	3.082	2.654
2	2.987	3.234	2.783
3	2.837	2.967	2.842
5	2.785	2.874	2.382
10	2.720	2.826	3.365

Table 6. Mass of salt crystals formed in the absence and presence of inhibitors ( $T = 120^{\circ}C$ , p = 2 bars)





Figure 8. NaCl crystals formed in a supersaturated solution treated with the product SA 2070 at 50 ppm

Figure 9. NaCl crystals formed in a supersaturated solution treated with the product AVA at 50 ppm

#### 4. Optimization test

The effectiveness of a treatment product cannot be evaluated only by the action of the product, but also by the amount of product used. The treatment concentration is of paramount importance from an ecological point of view, but especially from an economic point of view. Treatment products in the petroleum industry are generally quite expensive, so process optimization is a must for product selection. The use of a product effective at very high treatment concentrations can be expensive on the one hand and harmful to the environment on the other.

Experimental procedure: Prepare a series of washing solution by adding the salt deposit inhibitor to distilled water to obtain solutions at 2.5, 5.0, 10.0, 20.0, 30.0, 40.0, 80.0 and 100.0 ppm V/V. Prepare one liter of a supersaturated solution of 380 g/L of NaCl in an Erlen Meyer flask. Stir the solution and add the washing water dropwise until the crystals have completely disappeared at the bottom of the solution and note the volume of washing water added to the solution. The test was performed at room temperature of 20 ° C. The results of the test are given in the Table 7 and represented graphically in Figure 10.



Figure 10. Graphical representation of the results of the optimization test.(1) Volume of water required to dissolve excess salt in ml; (2) Percentage reduction in this volume required as a function of the treatment concentration.

The results show that the amount of wash water needed to dissolve excess salt crystals decreases with its concentration of treatment product. We have noticed that after the crystals disappear from the solution after adding the treated washing solution, the crystals gradually reappear over time when the solution is allowed to stand.

Optimization is about finding a compromise between the efficiency and economic considerations. According to the suppliers of the treatment products, and also according to some producers, a reduction in the volume of wash water by 40% within acceptable economic limits, would be profitable for the producer and beneficial for the environment.

Optimization of laboratory treatment, but under conditions which are closest to real conditions, will certainly offer substantial gains for the producer and less nuisance for the environment.

#### 5. Conclusions

At the end of this work, we can say that the three methods tested are based on the same criteria for evaluating the effectiveness of a water treatment product supersaturated with NaCl, namely, the reduction in the amount of crystals of NaCl formed, the morphology of these crystals and the way in which these crystals are linked to each other.

The first method - the one proposed by the supplier - seems to us the simplest, the fastest. However, the three allow us to judge the effectiveness of a product and to operate in the laboratory, a preselection of the most effective products. However, it is not easy to quantify the efficacy of the tested product, since a salt deposit inhibitor product can increase the solubility of salt in water, as it can act the tiny crystals to prevent them from spreading. grow and therefore cause blockages in the pipes. The same product can also have both actions simultaneously. Also, we noticed that the action of the tested products is limited in time, the effectiveness of a product must also take this parameter into account. If the oil-water separation facilities are far enough away from the injection point of the inhibitor product, the salt crystals can reform and settle before they reach the separator. Therefore, it would be better to remove the supersaturated water as quickly as possible, with the installation of FWKO (Free Water Knock Out) type separators at the head of the treated wells. This action will make it possible to avoid interventions on surface installations for salt plug extraction operations.

It is known that the effectiveness of a treatment product, whatever its nature, varies from one well to another and from one field to another. This behavior is linked to the chemistry of the medium and to the thermodynamic and hydrodynamic conditions of production.

This is why the evaluation tests must be carried out under conditions that come as close as possible to actual production or operating conditions.

The results of the optimization test that we carried out on the product considered to be the most effective, showed that the volume of wash water from salt wells can be reduced by 50% with a low consumption of treatment product. This reduction is synonymous with a reduction with a similar rate of free, emulsified or soluble hydrocarbon releases in nature.

The use of products that inhibit salt deposits for washing salt wells, in an optimized and rational manner, will certainly reduce the harmful impact on the environment and on the fragile ecosystems of the south of the country, inherent in the unbridled use of water in oil production, without constituting a heavy financial constraint for the producer.

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