Industrial RO Membrane Based Demineralization Plant Performance Optimization - An Innovative Low Level Silica Pretreatment Approach

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Abstract. India is a water-stressed country, having only 4% of the world's water resources available for 18% of the world's population. Multi-stage Reverse osmosis (RO) in combination with wastewater RO(WW-RO) is in practice now for the Industrial demineralization process actualizing the zero liquid discharge concept. The design of the de-mineralization plant depends upon the quality of the water source. Natural water holds a considerable amount of silica (SiO_2) up to 100 mg/L. The factors affecting RO membrane performance were studied, of which, silica fouling in combination with minerals and organics is found to be the main factor among other factors. The silica is hence complex in nature and silica fouling is generally not easy to control, thereby, resulting in performance failure of RO membranes. The present study discusses the effect of dolomite treatment, soluble magnesium compounds and sparingly soluble magnesium compounds on silica removal in boiler-feed water. It is found that the dolomite treatment in the pre-treatment step is ineffective for silica removal. The effective silica removal with soluble magnesium compounds increases the conductivity in the treated waters, and with sparingly soluble magnesium compounds is found only 40% effective; for increased effectiveness up to 86% pre-acidification was the condition which caused conductivity increase. In the present study, 90% of silica removal is achieved without pre-acidification along with the co-benefit of conductivity re-duction of feed water to the RO system. Therefore, the developed solution is efficient, cost-effective and environmentally friendly, which has not reported so far and is unclear in the literature available.

Keywords: MgO, Silica removal, Pre-treatment, Reverse osmosis Membranes.

1. Introduction

Globally, India ranks fifth in terms of electricity generation and sixth largest energy consumer with a growing energy demand of 3.6% per year due to economic development. The total electricity demand is projected to cross 950 GW by 2030[1]. India per capita water availability is declining rapidly due to factors like population growth, urbanization, industrialization, and climate change [2]. The National water consumption is expected to grow from 1.4% in 2025 to 9% by 2050 in power generation and this figure is likely to vary quite significantly from region to region[3]. In India, as per the business business-as-usual scenario, it is projected that the Industrial sector's Gross domestic product(GDP) contribution will be around 40% of the total GDP and

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the Industrial water demand is expected to increase by 102 m^3/person/year by 2050. The scarcity of water is already being faced by thermal power plants due to the non-availability of fresh water and this problem is expected to be further aggravated. Thus, there will be an urgent need to minimize water consumption requirements for thermal power plants.

Multi-stage Reverse Osmosis in combination with wastewater RO (WW-RO) presently is in practice for environment friendly industrial demineralization in line with the zero liquid discharge practices. Its design and efficiency depend upon the source water quality. Natural water sources hold silica (SiO_2) 1-40 mg/L and in some geographic areas, about 100 mg/L [4-7]. Due to silica's complex nature, silica fouling control is quite difficult, which often causes failure in RO system performance. The silica removal using soluble magnesium compounds was found effective, but they increased the conductivity in the treated waters. Therefore, the use of sparingly soluble magnesium compounds was found to be a promising alternative but only 40% silica removal was obtained, for higher Silica removal of 80-86% pre-acidification was needed [8]. In the present study, effectiveness of silica removal is found 90% without pre-acidification with the additional benefit of reduced feed conductivity to RO as suggested elsewhere[9]. Commercial use of powerful cleaning chemicals like hydrofluoric acid (HF) and ammonium bifluoride (NH_4HF_2) generally in practice to control silica fouling, but can cause equipment, membrane damage and environmental concerns [10]. Due to fouling, the recovery rate reduces and demands high feed water and more reject generation for the equal water treatment volume. This may further cause inefficient running and sometimes cause complete failure of the RO system. Since the raw water source available is hard water of a high pH around 8.5, the calcium-induced silica fouling is also one of the possible reasons for failure in the performance of RO membranes in line with earlier findings, Severe colloidal silica fouling was also observed during the trial using hardwater, evident by more rapid flux decline, more colloidal silica on scaled membrane in autopsy and an increased amount of silica from dissolving scaling species on scaled membranes surfaces[11]. The mineral scaling issue is also seen in RO membrane, found responsible for low water recovery rates and deteriorated system performance, Gypsum scaling causes pore wetting by crystallization mechanism silica scaling forms highly adhesive, irreversible layers, by polymerization mechanism [12]. The relationship between membrane surface properties and silica fouling in RO is also proven where the strong relationship of silica scaling with the membrane surface charge was seen [13]. Demineralization plays a crucial role in preventing silica entry to the boiler for steam generation, the elevated silica concentrations in steam can lead to silica deposition in the low-pressure turbine. Also, a thin layer of silica scale in the boiler can cause boiler tube failure and result in huge economic loss to the power plant.

The higher water treatment cost of is affordable in comparison to efficiency loss due to scale deposition in water wall tubes resulting in low heat transfer, and high fuel consumption which may further lead to boiler tube failures and hence forced shutdown of power plant unit. The boiler tube with iron and silica scale is shown in Fig.1 and the composition of the scale is shown in Table 1.

Fig.1. The picture showing boiler tube blockage with predominantly iron and silica rich deposit.

Table-1 Chemical Analysis of the boiler tube blockage material

Sample Name	% Fe as Fe_2O_3	% Cu as CuO	% Ca as CaO	% Na as Na_2O	% Mg As MgO	% K As K_2O	% Si As SiO_2
Deposit sample	65.4	1.49	0.13	0.46	0.15	0	25.2

Even small-scale boiler of 500hp can have 2% energy loss with calcium and magnesium scale of 0.8mm and same thickness scale of iron and silica the energy loss is multiple times i.e. 7%[14].

1.1 Background

Agricultural development has exponentially increased in recent times near the power plant study area, resulting in increased organic loading in source water[Table 2]. The presence of organics in water along with silica can accelerate the silica fouling in RO membranes. Silica fouling against organic content loaded in feed water was an added research challenge[15]. The same has been seen elsewhere in autopsy results of high Loss on Ignition (LOI) value along with the presence of silica and other metals. The foulant composition had organic matter and inorganic matter, viz., iron, calcium, magnesium, acid insoluble matter, silica, sulphate, molybdenum, boron, potassium, sodium, hydrocarbons, total phosphate, organo-phosphate, ortho-phosphate. Simultaneously, minor contaminants like soluble organic fouling, chromium, aluminum, manganese, sulphite, cobalt, copper, nickel, zinc, and fluoride have shown their adequate presence. The introduction of sodium hypochlorite (hypo) dosing in combination with ClO_2 in clarifier helped us in controlling organics.

After pretreatment, the demineralization plant includes the ultrafiltration-reverse osmosis-mixed bed (UF-RO-MB) system. In the RO systems, two key parameters of permeate (silica and conductivity) were showing signs of deterioration over time and indicative of several factors that could be impacting the overall efficiency and lifespan of the membranes[Fig-2&3]. The RO permeate conductivity and silica were found to be touching 80 µS/cm and 8500 µg/L against design estimates of 32.47 µS/cm and 286 µg/L respectively. The parameters are found to show an increasing trend from the start of the RO plant operation. Since the commissioning, the design raw water reactive silica was 20 to 23 mg/L as SiO_2 and the silica in the clarifier outlet of the pre-treatment plant was maintained 18-19 mg/L against the design value of 10 mg/L[Table-3]. At the ultrafiltration (UF) outlet, the SDI values were touching five against the design value of maximum 3 where SDI parameter T1 was found around 50 seconds against the recommended value of 20 seconds, indicating issues in the pre-treatment step and resulted in forced shut down of UF-RO-MB demineralization plant.

Table-2: Total Organic Carbon (TOC) measurement data**.**

Sample Name	TOC (mg/L)
Raw Water	6.974
Aerator O/L	6.527
Clarifier O/L	5.984
Filter bed O/L	5.744
UF I/L	5.850
UF O/L	4.643
RO Permeate	0.113
RO Reject	107.9

Table-3: Reactive silica Value variation in DM Process.

Sample Name	Reactive silica(mg/L)
Raw water	20-23
Clarifier outlet	18-19
RO inlet	16-18
RO permeate	0.9-8.5
RO reject	102-58
MB outlet	0.007-0.012

Investigation and root cause analysis were performed, and several process modifications were made like at Pretreatment, the dosing points were shifted to near aerator to have enough opportunity for mixing and reaction time from earlier dosing point at clarifier, scrapper drive of DM clarifier under continuous operation, the design oxidation-reduction potential(ORP) was revised from 100 to 200 and recommended to keep below 200, optimized dosing of ClO_2 along with sodium hypochlorite, maintaining the UF & RO cartridge pre-filter dP below 0.5 kg/cm2 and the chemical enhanced backwash frequency of UF has been doubled given fouling, however, integrity of UF membrane was found ok, but improvement in SDI was observed for shorter duration only.

It was noticed that UF was having periodical differential pressure(dP) high for which backwash frequency has been doubled, the rise of dP from about 0.5 kg/cm2 to 0.9 kg/cm2 was unusual. The main cause for this was supposed to be iron, turbidity and organic fouling, The raw water TOC is seen to be more than 7 mg/L. It was found that UF is not at all preventing the passage of colloidal silica which it was expected to do. Some membranes were extracted earlier for autopsy analysis at another plant with a history of RO efficiency deterioration, the analysis revealed the type of foulants that have clogged the membranes, thus easing root cause analysis and prevention in the future. As per the report, the main foulants were silica, colloidal silica, phosphate, and iron. The sample elements were taken from the front and rear ends of RO stage 1. The visual observation showed the fouling, the front one was more fouled showing iron and colloidal fouling were predominant factors. This also con-firmed the initial assumptions. The probe test and its analysis revealed many of the membranes were irreparably damaged. Also, there was a problem of flow balancing, among membranes. The use of silica-based anti-scalant for high reactive silica at the RO inlet was started, however, no improvement in permeate parameters was observed.

The UF & RO membrane's performance so deteriorated that it was not economical to use them further for water de-mineralization. Therefore, the replacement of both UF & RO membranes was done. Even after the replacement of the new UF & RO membrane, the SDI was maintained just below the manufacturer's SDI limit of five but not below three and the initial time(T1) parameter of SDI was also found more than the recommended value of 20 seconds. The RO permeate silica was found to be multiple times (more than 1 mg/L) than the design (0.3 mg/L) due to high input silica (>10 mg/L) in the RO feed with the existing dolomite treatment. The high silica in RO permeates further reduces the output of MB and results in high chemical consumption and, short operational life of resin and equipment. Hence, improvement of the pre-treatment process along with silica reduction below ten mg/L is the key factor of performance improvement of the DM plant.

Mitigating silica scaling in subsequent RO systems, the silica concentration reduction in the feed water during the pre-treatment step and the remaining particle removal in UF can be a simple but effective step. In this section, experiments were done to mitigate the silica issue of the pretreatment-demineralization (PT-DM) system. In the present case, the conventional dolomite treatment was seen to be ineffective for silica removal and contributed as a primary factor in the performance deterioration of RO membranes. The effective silica removal with soluble magnesium compounds increases the conductivity in the treated waters while a promising alternative of using sparingly soluble magnesium compounds can achieve only 40% silica removal; for higher Silica removal of 80-86%, pre-acidification was needed. In the present study, 90% of Silica removal is achieved without pre-acidification along with the co-benefit of conductivity reduction of feed water. [16]. The result obtained with sparingly soluble magnesium compound was not only effective for silica removal but also decreased the conductivity of pre-treated water significantly, Therefore, the developed low-level silica reduction along with conductivity reduction in pretreatment step of RO-based Demineralized water treatment plant is efficient, cost-effective and environment friendly, which is not covered so far and unclear in the literature available [17-18]

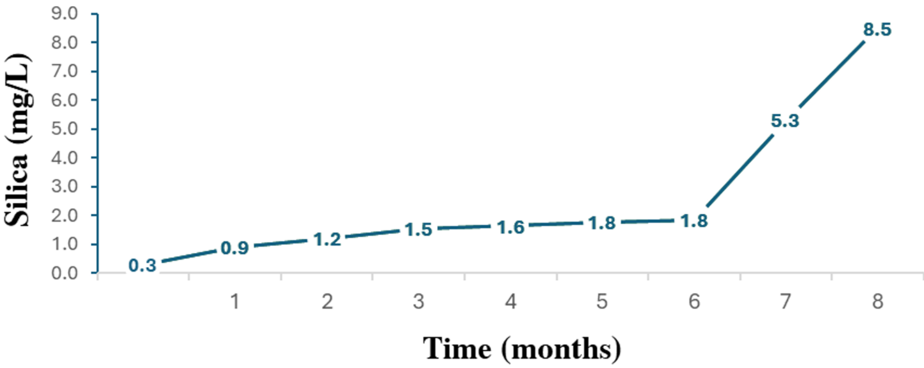

Fig.2. The performance deterioration of RO with respect to permeates silica.

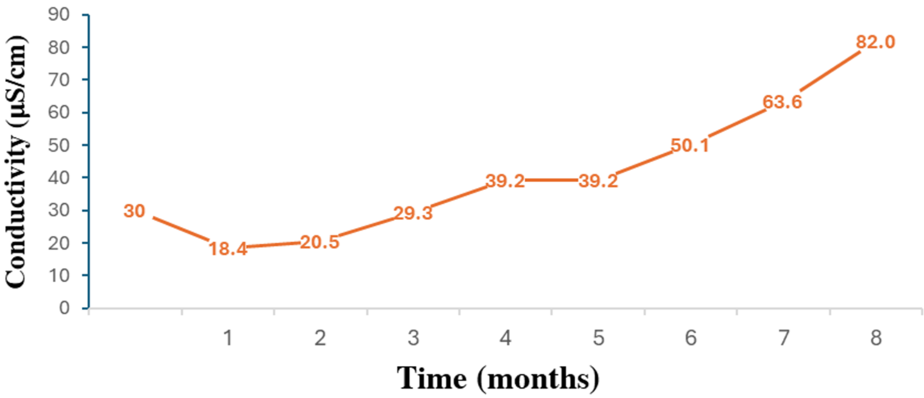

Fig.3. The performance deterioration of RO versus permeates conductivity.

1.2 Chemistry of Silica

The chemistry of silica (SiO_2) is complex, silica has four covalent bonding sites like carbon. The silica nucleus is bigger as compared to the nucleus of carbon, double or triple bond formation is not common in silica, also more than six silica atoms long chain compounds are not common. silica exists in different crystalline as well as amorphous forms. In water, the solubility of crystalline silica is exceptionally low in the order of six mg/L SiO_2. Amorphous silica solubility of 100-140 mg/L has a much higher SiO_2. When silica is dissolved in water, it forms Mono silicic acid ($Si(OH)_4$) which monomeric will remain in the state if its concentration stays less than about 2 mM [19]. In comparison to carbonic acid Silicic acid is a weak acid with a smaller Ka than carbonic acid. Silicic acid K1 = $2x10^{-10}$, silicate ions can be found only a few µg/L at neutral pH, but the presence of alkalinity encourages the formation of silicate ions, increasing the solubility of "silica"[Eq.(1)].

$$Si(OH)4 + OH^{-} \rightarrow [SiO(OH)3]^{-} + \mathrm{H2O} \tag{1}$$

With the increase in pH, silica solubility increases and at pH around ten, silica is found in the form of silicate ions and is soluble, which is found in the present study too. In the absence of alkalinity or other stabilizing ions, Silicic acid molecules polymerize easily as ($H2SiO_3$)n. Silica is one of the most common scales on RO membranes during desalination processes. The silica exists in three forms firstly Monomer silicic acid ($Si(OH)_4$), which is also known as dissolved or reactive silica, secondly colloidal or polymer silicic acid, and thirdly particulate silica as clay, silt, and sand. Colloidal silica does not react with ammonium molybdate and is referred to as non-reactive silica [20-21].

Table-4 Different type of Silica with Size [23]

Silica form	Size(microns)
Filterable	>0.45
Colloidal	0.01-0.45
Polymeric	<0.01
Monomeric	<0.0001

The particles below 0.1 microns are likely to exist in dissolved form. The colloidal silica can appear after digestion like in a power plant boiler drum at high pressure and temperature. The filterable silica, less than 0.45 microns can be evaluated using a 0.45- micron filter to find the silt density index (SDI) ahead of RO systems [Table 4]. Silica solubility is of important concern as it gets concentrated in the reject stream of the membrane. RO systems cannot achieve complete removal of reactive silica-like ion exchange but are far better in removing non-reactive silica like colloidal as well as particulate silica

1.3 Chemistry of Magnesium oxide & Silica

Magnesium is the eighth most abundant element in the Earth's crust (about 2.3% by weight) and the third most abundant element in seawater solution (about 1300 mg/L), present naturally as dolomite, magnesite, and silicate in rocks. The market volume of magnesium oxide (MgO) is estimated to be around 22.6 million metric tons by 2030[22]. The light burnt or calcined MgO has high reactivity and a large specific surface area. The large surface area and proximity to silica molecules allow MgO to be much more effective as found in the present case. The advantage of calcined MgO is that it adds almost no dissolved solids to the water. pH and temperature are also key factors in silica reduction by precipitation. The precipitation increases with increasing temperature and completes at elevated temperatures. pH kept high enough i.e., Higher than ten to result in precipitation of magnesium but controlled in such a way that not to go so high i.e., more than eleven which can make the precipitant resoluble.

$$\mathrm{MgO} + \mathrm{SiO2} \xrightarrow{\mathrm{pH}\sim 10} (\mathrm{MgO})\mathrm{x}\,(\mathrm{SiO2})\mathrm{x}.(\mathrm{H2O})\mathrm{x}\ \ (\mathrm{x} = \mathrm{variable}) \tag{2}$$

Silica removal by magnesium hydroxide is not clear and may be simple adsorption onto the surface of $Mg(OH)_2$ and removed to a certain extent with concurrent precipitation with $Mg(OH)_2$ or Magnesium silicate may be formed [e.g., Mg_2SiO_4 (Forsterite)]. However, Magnesium-based compounds along with Coagulants and Flocculants are found quite effective in promoting the aggregation of small particles into larger flocs and therefore lead to clear water at the outlet of the clarifier[Eq.(2)]. The clarity of the water at the upper side of the precipitated flocs was monitored and compared with the different test jars. The jar having the clearest water shows the most successful treatment conditions [23-25].

2. Experimental

2.1 Materials and methods

The Indian standard 3025-50 (2001): methods of sampling and tests (Physical and Chemical) for water and wastewater, part 50: Jar Test (Coagulation Test) was used to determine the optimum dosage of coagulant, coagulant aids and the optimum pH of coagulation for removal of turbidity and color caused by colloidal and non-settleable particles followed by sedimentation under quiescent condition.

The instrument used for this study consists of a semi-automatic jar test apparatus Scientech make, model SE-157 supplied by ISO 9001 certified M/s Scientech, India,

The jar test apparatus used has an LED digital display, a touch-sensitive keypad, six jars, and six stainless steel stirring paddles with a 10-300 rpm speed range along with a speed regulator and timer that supports a determination of the exact amount of input needed to trigger flocculation.

Standard methods for the examination of water and wastewater, APHA 24th edition, 4500-H^+ pH, 2510-conductivity, 2130-turbidity, 4500-SiO_2 silica, method 3111D were used to determine the parameters including reactive as well as colloidal silica.

The material used during the jar test experiment is optimized as calcined MgO, hydrated lime, ferric chloride and anionic polyelectrolyte(PE) (Table 5).

Table-5 Material used for jar test study

Material	Grade	CAS No.
$FeCl_3$	IS 711:1970(2006)	7705-08-0
Hydrated Lime	IS 1540:1990(2003)	1305-62-0
Dolomite	IS 1760-3 (1992)	16389-88-1
PE	Industrial grade	9003-05-8
Calcined MgO(light)	Industrial grade	1309-48-4

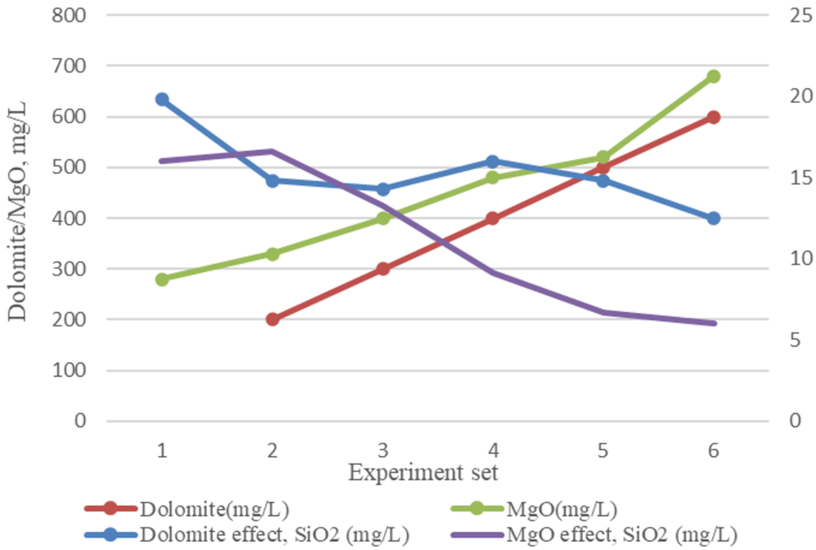

Fig.4. Dosing optimization of dolomite and MgO for silica reduction

Various experiments were performed to establish the optimum combination of Dolomite as well as MgO, but even after using up to 600 mg/L dolomite silica reduction could not achieve the design requirement of less than 10 mg/L silica at the feed of RO, however, it could be achieved with only 480 mg/L MgO [Fig.4].

Jar tests with various combination has been performed which simulate the similar conditions of mixing and settling present in clarifiers of pre-treatment plants with the proper control of stirring speed[Table-5]. The 480mg/L has been found the optimized dose of MgO for required silica reduction of less than 10mg/L, while with dolomite, it could not be achieved

Table-5 The comparison of jar test results obtained versus raw water quality

Parameters/Set	Set-1	Set-2	Set-3	Set-4	Set-5	Set-6
Dolomite(mg/L)	200	300	400	500	600	700
MgO(mg/L)	280	330	400	480	520	680
SiO_2 (mg/L), Dolomite effect	14.8	14.3	16	14.8	12.5	17.4
SiO_2 (mg/L), MgO effect	16	16.6	13.3	9.1	6.7	6

3. Result & discussion

The pre-treatment of river water is an essential integral step to producing quality DM water. The design input silica requirement for RO based DM plant was 10 mg/L, the existing dolomite-based treatment combination of dolomite, FeCl3, lime and poly-electrolyte (PE) was found to be ineffective and not able to reduce the silica level at the desired design level whereas the newly developed treatment methodology MgO, $FeCl_3$, lime and PE can address the silica issue and found to be promising silica treatment option for RO based DM plant. The co-benefit of the developed method is not only found promising in the removal of reactive silica, but the more problematic colloidal silica is also reduced quite significantly. The other advantage of the method is a reduction in Conductivity which further reduces the ionic load on RO membranes and hence enhances life and performance of the RO-DM system.

The Jar test results has been compared for both dolomite as well as MgO under similar conditions of mixing and settling present in clarifiers of pre-treatment plants with the proper control of stirring speed[Table-6]. The material used during the jar test experiment is optimized as MgO 480 mg/L, Lime 120 mg/L, $FeCl_3$ 30 mg/L and PE 2 mg/L.

Table-6 The comparison of jar test results obtained versus raw water quality.

Parameters	Raw water	Design Dolomite treatment	Newly developed treatment
pH at 25°C	8.8	10.14	10.04
Sp. Cond(μS/cm)	451	358	357
Turbidity (NTU)	1.60	0.2	0.1
Total SiO_2 (mg/L)	32.1	14.2	3.20
Colloidal SiO_2 (mg/L)	12.0	1.3	1.0
Reactive SiO_2 (mg/L)	20.1	12.9	2.2
Silica reduction (%)		56%	90%

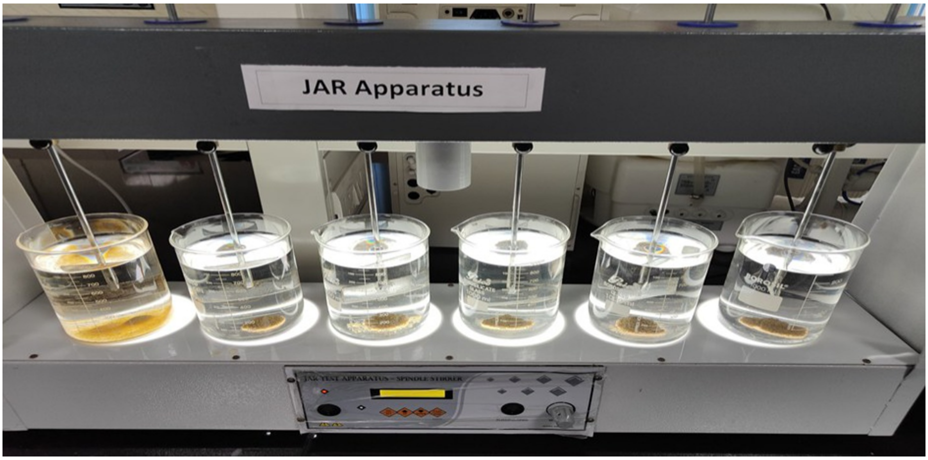

Fig.5. Test setup shows clear water after test completion. (most clear water in right most jar of optimized Mg based treatment)

It can be seen in the above results that the dolomite for silica treatment in combination with other OEM recommended pre-treatment plant (PTP) chemicals are found to be only 56% while Mg-based compound with the same other OEM recommended PTP chemicals were found to be most effective in treating reactive as well as colloidal silica. Lab simulation efforts were started to find primary factors, interrelation of constructive interaction among factors, if any, and to discover any other controlling factors that might exist to make pretreatment at WTP more effective. The design of the experiment (DOE) initially constituted two factors with dolomite, $FeCl_3$, PE and lime as originally designed by specification[Fig.5]. When this did not reach the desired result, another substitute for dolomite chemical was found and a three-factor with three-level experiment was planned. In total, more than twenty experiments were performed at the lab. Finally, the best formulation of substitute chemicals was found to produce acceptable results as per the goals set out in the planning stage. The most noteworthy was the finding that simply replacing dolomite with another suitable substitute keeping all other dosing chemicals constant, could reduce both iron, TOC, and colloidal best through a three-factor constructive interaction. The proposed pre-treatment is effective in reducing both forms of silica, i.e., reactive, and colloidal silica.

The developed solution is a key step toward mitigating the challenges associated with the pre-treatment of silica in the DM-RO plant.

1. Realistic assessment of designed dolomite treatment
2. Ascertained the factors towards UF-RO deterioration.
3. Effectively running the Clarifier with optimized chemical dosing.
4. Ascertained the reasons behind design silica carryover in the pre-treatment step.
5. Developed a method for obtaining the Silica level below the design value for the RO inlet.

4. Conclusion and Recommendations

The factors affecting RO membrane performance were studied, the mineral and organics-induced silica fouling is found to be the main factors among other factors. The silica is complex, and its fouling is not easy to control, which results in the performance failure of RO membranes. Mitigating silica scaling in subsequent RO systems, the silica concentration reduction in the feed water during the pretreatment step and the remaining particle removal in UF can be a simple but effective step. In the present study, various Jar test experiments were performed to establish the optimum combination of dolomite as well as MgO, but even after using up to 600 mg/L dolomite silica reduction could not achieve the design requirement of less than 10 mg/L silica at the feed of RO, however, it could be achieved with only 480 mg/L MgO. The key finding of the proposed pre-treatment is that it is effective in reducing both forms of silica, i.e., reactive, and colloidal silica and 90% silica removal is achieved along with the co-benefit of conductivity reduction without the pre-acidification requirement as suggested elsewhere. Therefore, the developed solution is efficient, cost-effective and environmentally friendly, which has not been reported so far and is unclear in the literature available. In NTPC, the treatment is its first of a kind and can be replicated in the PT-UF-RO-MB demineralization plants operational at many of its thermal power plants for further efficiency improvement.

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