# STRATEGIC NANOFILTRATION APPLICATIONS IN CERAMIC AND RELATED INDUSTRY WATERS

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## ABSTRACT

Phenomena such as climate change, world population growth, and industrial development have significantly affected both the quantity and quality of currently available water resources. In industry in particular, where a considerable volume of wastewater is generated, the search for and application of new treatments such as membrane technologies, which open up new possibilities with regard to the valorisation of this key resource in view of threatening water scarcity, are of great interest.

This study sets out some of the results obtained after several years' work on nanofiltration technologies and their application in the treatment of liquid effluents in different ceramic industry applications. The following water matrices were used in the work: industrial wastewater and supply water. In all cases, the application of nanofiltration through polymer membranes was studied.

In the wastewater, the target pollutants were as follows: boron, organic compounds, and inorganic ions. For the boron, the retentions obtained ranged between 75 and 90%. The reduction achieved for the chemical oxygen demand (COD) was above 80% and, with regard to conductivity, reductions exceeding 90% were obtained. With regard to the supply water, the study focused on the removal of divalent electrolytes, in particular $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$. Retentions close to 100% were obtained for $SO_4^{2-}$ and between 60% and 90% for $Ca^{2+}$ and $Mg^{2+}$, depending on the membrane used.

A further characteristic of interest in nanofiltration processes, provided the efficiencies achieved are those technically required, is the cost per cubic metre treated water. In the studied cases, this was lower than that of competing techniques such as reverse osmosis (RO).

## 1. INTRODUCTION

Water is a vital resource for life and for the economy. However, this resource is facing many serious challenges, such as water scarcity and the pollution and degradation of ecosystems. Recent studies have shown that in 2050 there will be hydrological river basins with severe water stress. Therefore, additional pressures could emerge and toughen the competition between different water users, such as agricultural irrigators, ecosystems, cities, and industries [1].

Industry, in general, is a major water consumer and wastewater producer. In the particular case of the European ceramic industry, since it is localised and concentrated in clusters, the environmental impacts related to water use are of great significance. Consequently, the search for and application of new treatments such as membrane technologies could open up new possibilities with regard to the valorisation of water resources, reducing water emissions and optimising water use.

The ceramic BREF [2] sets out several wastewater treatment systems that are applicable to the sector. However, the most widely applied cleaning systems are based on physico-chemical treatments. These treatments effectively remove suspended solids and certain metals, but they do not remove a number of wastewater pollutants such as boron, organic compounds, and dissolved inorganic ions. These pollutants prevent discharge of the clarified water, as well as its reuse in production process stages other than floor cleaning or slurry preparation.

For the removal of such pollutants that tend to resist currently implemented treatments, it is necessary to resort to tertiary treatments which, though more expensive, can provide the water with sufficient quality to enable it to be reused in certain, currently unimaginable ways.

The different tertiary treatments at issue include membrane filtration treatments and, in particular, nanofiltration (NF). Generally, NF is defined as a technology that lies between ultrafiltration (UF) and reverse osmosis (RO). It is more effective than UF because of its capacity to retain salts, and it is more selective than RO. It therefore achieves optimum results for applications where such high efficiencies as those provided by RO are not required, with considerably smaller energy consumption [3].

Examples of nanofiltration applications include the following: water softening, pre-treatment for subsequent desalination processing, removal of micro-pollutants for water purification and reuse, extraction and separation of salts and solutes of low molecular weight. Nanofiltration is being applied in a wide range of industries. A recent novel application is, for example, the colorants industry in which this technique is used as a treatment process to concentrate certain effluents or products [4].

## 2. OBJETIVE

The main objective of this study is to provide a comprehensive view of the possibilities that nanofiltration techniques can provide in handling the industrial water generated in the ceramic sector, ranging from process water to produced wastewater, increasing savings in consumptions and enhancing company sustainability.

## 3. METHODOLOGY

### 3.1. TYPE OF WATER TO BE TREATED

Two types of water were considered:

#### MATRIX 1. Process water

The different uses of water in the ceramic industry determine its ultimate destination.

| Type of use | Process stage |
|---|---|
| Raw material | Preparation of ceramic compositions |
| Coolant | Pressing, machining of ceramic tiles, maintenance, frit quenching, etc. |
| Steam generation | Cogeneration systems (boilers) |
| Washing | Raw materials preparation, glaze preparation and application, and treatment of gas streams |

*Table 1. Water uses and process stages*

#### MATRIX 2: Industrial wastewater

Much of the water used in production in the ceramic industry does not become wastewater, as it is evaporated in the drying and firing processes. In other cases, e.g. when it is used as heat exchanger, the water is contained in closed circuits and, except for incidental purges, will not be transformed into wastewater either.

In this sector, the cleaning operations in the glaze preparation and applications sections generate the most wastewater. The wastewater chemical composition is characterised by the presence of suspended solids, dissolved anions, dissolved and/or suspended heavy metals, more or less variable quantities of boron, and organic matter.

The concentrations of these elements depend on the type and composition of the glazes involved and on the volume of water used in the cleaning stages, cleaning generally being manual [5].

## 3.2. STUDIED POLLUTANTS

The following pollutants were studied:

- **Boron:** Boron compounds are used as raw materials in a great many industrial processes, including the ceramic industry [6]. Boron is quite often present in ceramic industry wastewater and the boron content can range from 1 to 80 mg/l. Its presence in treated water is due to boron's high solubility and small ion size, which make it difficult to remove with the most common treatment techniques used in the ceramic sector.

- **Divalent inorganic salts:** The high ionic conductivity associated with the presence of inorganic salts prevents reuse of the wastewater. Even in clean process water, high electrolyte concentrations considerably reduce production process efficiency. In ceramics in particular, divalent electrolytes are highly flocculating, making it necessary to prevent this effect by adding deflocculants in the preparation of ceramic suspensions for the production of the ceramic body, as well as in the preparation of glaze compositions and additives [7].

- **Organic matter:** The organic matter in ceramic industry wastewater comes mainly from screen printing vehicles and other substances used in ceramic tile decoration. The polymers used in the flocculation process in the cleaning stages are a further source of organic matter. The decomposition of organic polymers can lead to problems of smells when the physically and chemically treated water is used in cleaning operations. On the other hand, the presence of organic matter (raw material in glaze preparation) in process water can give rise to rheological problems, making water reuse practically unfeasible.

## 3.3. MEMBRANES

Nanofiltration (NF) is the membrane filtration technique that has undergone the greatest development in recent years, above all in industrial environments, owing to its high selectivity. Nanofiltration is characterised by the following distinguishing features, compared to other techniques: (i) a very high multivalent ion retention owing to charge effects; (ii) a neutral solute retention that depends on the size and shape of the solute, with the solutes being retained by steric effects.

In this study, DOW FILMTEC membranes were used. In the laboratory-scale tests, flat membranes were used, whereas in the pilot plant tests, industrial spiral wound membranes were used.

The specifications provided by the manufacturer for the two membranes used are detailed in Table 2.

| Membrane | Permeate flow (L/(h $m^2$ bar)) | $MgSO_4$ retention (%) |
|---|---|---|
| NF-90 | 8.68 | >97 |
| NF-270 | 10.68 | >97 |

*Note: Permeate flow and retention are based on the following working conditions: 2000 ppm $MgSO_4$, 25°C, and 15% recovery. The permeate flows for individual elements can vary between -15% and +50%*

*Table 2. NF membrane specifications provided by the manufacturer*

## 3.4. EQUIPMENT

The study was conducted on a laboratory scale and, wherever possible, a scale-up was performed to a pilot plant or semi-industrial scale. In each case, depending on the dimensions of the trial, the equipment best suited to the proposed scale-up was used.

The equipment used is shown in the following figures:

Para la elaboración de este estudio se han realizado trabajos a escala de laboratorio y en los casos que ha sido posible, se ha realizado un escalado a planta piloto o semi-industrial. Para cada uno de los casos, según las dimensiones de la prueba, se ha utilizado el equipamiento que mejor se adaptaba al escalado propuesto.

En las siguientes figuras se muestran el equipamiento utilizado:

*Figure 1. Laboratory-scale equipment*

*Figure 2. Pilot plant facility*

Independently of the scale involved, all the equipment contained the following common elements: feed tank(s), circulation pump(s) or system applying a pressure differential, pressure gauges for pressure control, flow meters, thermometers, membrane housing modules, sampling systems, and data logging systems. In the case of the pilot plant, there was a pre-treatment system with microfiltration ceramic membranes to extend the service life of the polymeric nanofiltration membranes.

### 3.5. PROCESS

The experimental procedure followed during the tests conducted with the membranes is detailed below:

- Conditioning of the membrane
- Determination of membrane permeability to distilled water.
- Measurement of the flow and retention for several real waters.

Retention calculation: the samples collected in the previous phase were analysed and the results were compared with the analyses of samples of the feed solution. The retention was calculated from the following equation:

$$R = (1 - \frac{C^{permeado}}{C^{alimento}}) * 100$$

Where $C^{permeado}$ and $C^{alimento}$ are the permeate and the feed solution concentration, respectively.

### 3.6. ANALYSIS

The conductivity and pH of the waters were measured using a JF 340 WTW conductimeter and a PHN 81 Tacussel pHmeter, respectively. The methods used in analysing the different parameters and species in the test waters are listed in Table 3.

| Parameters | Method |
|---|---|
| DISSOLVED ORGANIC CARBON (DOC) | **COMBUSTION–NON-DISPERSIVE INFRARED** |
| B | Azomethine-H method by UV-VIS spectrophotometry |
| $SO_4^{2-}$ y $Cl^-$ | Ion chromatography |
| $Na^+$, $Ca^{2+}$ y $Mg^{2+}$ | **ATOMIC ABSORPTION SPECTROPHOTOMETRY UNE 77-056** |

*Table 3. Methods and equipment used in analysing the industrial waters*

## 4. RESULTS AND DISCUSSION

The results obtained in each studied case are presented in the form of data sheets that detail the most noteworthy information:

<table>
<tr><th colspan="4">Specific objective</th></tr>
<tr><td colspan="4">Removal of organic matter from ceramic tile manufacturing wastewater.</td></tr>
<tr><th colspan="4">Origin of the water</th></tr>
<tr><td colspan="4">Wastewater produced in the company after physical and chemical treatment.</td></tr>
<tr><th colspan="4">Membrane used</th></tr>
<tr><td colspan="4">NF-90</td></tr>
<tr><th colspan="4">Results of the parameters removed</th></tr>
<tr><th>Parameter</th><th>$C^{Feed}$</th><th>$C^{Permeate}$</th><th>Retention (%)</th></tr>
<tr><td>DOC (mg/L)</td><td>18-456</td><td>3-81</td><td>82-84</td></tr>
<tr><td>Conductivity (µS/cm)</td><td>1520-7260</td><td>270-2350</td><td>68-82</td></tr>
<tr><td>Boron (mg/L)</td><td>3-12</td><td>2,8-11</td><td>9-10</td></tr>
<tr><td>Sodium (mg/L)</td><td>208-1228</td><td>42-417</td><td>66-80</td></tr>
<tr><td>Calcium (mg/L)</td><td>130-22</td><td>1,2-1,3</td><td>99</td></tr>
<tr><th colspan="4">Membrane used</th></tr>
<tr><td colspan="4">NF-270</td></tr>
<tr><th colspan="4">Results of the removed parameters</th></tr>
<tr><th>Parameter</th><th>$C^{Feed}$</th><th>$C^{Permeate}$</th><th>Retention (%)</th></tr>
<tr><td>DOC (mg/L)</td><td>18-456</td><td>3-140</td><td>30-84</td></tr>
<tr><td>Conductivity (µS/cm)</td><td>1520-7260</td><td>1100-5800</td><td>20-28</td></tr>
<tr><td>Boron (mg/L)</td><td>3-12</td><td>2.8-10</td><td>13-14</td></tr>
<tr><td>Sodium (mg/L)</td><td>208-1228</td><td>128-1032</td><td>16-38</td></tr>
<tr><td>Calcium (mg/L)</td><td>130-22</td><td>6-56</td><td>57-71</td></tr>
<tr><th colspan="4">Uses of the treated water</th></tr>
<tr><td colspan="4">The organic matter content decreased considerably; depending on the membrane used, the electrolyte concentration also diminished significantly. The treated water met quality criteria for use in:<br>• Cleaning operations.<br>• Glaze and engobe preparation: the trials conducted were positive</td></tr>
<tr><th colspan="4">Observations</th></tr>
<tr><td colspan="4">At high DOC concentrations, the first membrane provided better efficiencies. At low concentrations, membrane efficiencies were similar. Nanofiltration is a tertiary cleaning system and needs to be preceded by physical and chemical treatment and microfiltration.<br>When the membranes are not running, they need to be kept clean and disinfected (sodium bisulphite).</td></tr>
</table>

*Table 4. Removal of organic matter and electrolytes from wastewater. [8]*

<table>
<tr><th colspan="4">Specific objective</th></tr>
<tr><td colspan="4">Removal of boron and electrolytes from third-fire ceramic tile production wastewater.</td></tr>
<tr><th colspan="4">Origin of the water</th></tr>
<tr><td colspan="4">Wastewater produced in the company after physical and chemical treatment.</td></tr>
<tr><th>Membrane used</th><th colspan="3">Working conditions</th></tr>
<tr><td rowspan="2">NF-90</td><th>P (bar)</th><th colspan="2">Permeability (L/hm$^2$bar)</th></tr>
<tr><td>5-7</td><td colspan="2">2-4</td></tr>
<tr><th colspan="4">Results of the removed parameters</th></tr>
<tr><th>Parameter</th><th>$C^{Feed}$</th><th>$C^{Permeate}$</th><th>Retention (%)</th></tr>
<tr><td>DOC (mg/L)</td><td>12-19</td><td>2.8-9</td><td>47-82</td></tr>
<tr><td>Conductivity (µS/cm)</td><td>1025</td><td>5.7</td><td>99</td></tr>
<tr><td>Boron (mg/L)</td><td>282</td><td>5</td><td>98</td></tr>
<tr><td>Sodium (mg/L)</td><td>834</td><td>13</td><td>98</td></tr>
<tr><td>Calcium (mg/L)</td><td>69</td><td>0,5</td><td>98</td></tr>
<tr><th colspan="4">Uses of the treated water</th></tr>
<tr><td colspan="4">The treated water quality was appropriate for use in:<br>• Cleaning operations.<br>• Glaze and engobe preparation: the trials conducted were positive.<br>• The treated water could be discharged to a public wastewater treatment plant (WWTP) provided the set discharge limit of 3 mg/L boron was not exceeded.</td></tr>
<tr><th colspan="4">Observations</th></tr>
<tr><td colspan="4">To achieve higher boron removal efficiencies, the pH of the water to be treated needed to be modified. This enabled boron retentions above 80% to be achieved.<br>As noted previously, nanofiltration is a tertiary cleaning system. In this case, it therefore needs to be preceded by physical and chemical treatment, in addition to microfiltration to extend membrane service life.<br>As wastewater is involved, when the system is not running, the membranes must always be kept clean and disinfected with sodium bisulphite.</td></tr>
</table>

*Table 5. Removal of boron and electrolytes from wastewater*

| Specific objective | | |
|---|---|---|
| Conditioning of clean process water for additives production. Removal of divalent electrolytes from process water. | | |
| **Origin of the water** | | |
| Clean groundwater supplied from a well. | | |
| **Membrane used** | **Working conditions** | |
| | **P (bar)** | **Permeability (L/hm²bar)** |
| NF-270 | 5.5-7 | 5.6 |

| Results of the removed parameters | | | |
|---|---|---|---|
| **Parameter** | **$C^{Feed}$** | **$C^{Permeate}$** | **Retention (%)** |
| Conductivity (μS/cm) | 1200-1450 | 398-900 | 67-38 |
| Chlorides (mg/L) | 71-96 | 40-84 | 13-44 |
| Sulphates (mg/L) | 303-311 | 5,4-57 | 82-98 |
| Sodium (mg/L) | 46-47 | 22-35 | 24-53 |
| Calcium (mg/L) | 137-198 | 21-100 | 50-85 |

**Uses of the treated water**

The treated water could be used in processing ceramic additives, with the following advantages:

- Improvement of the characteristics of the products fabricated.
- Possible replacement of reverse osmosis treatments used in water conditioning:
  - Decreased retentate quantity
  - Greater selectivity in divalent electrolyte separation.
- The retentate could be discharged to a public wastewater treatment plant (WWTP) provided the set discharge limit of 3000 μS/cm conductivity was not exceeded.

**Observations**

In this case, although the water to be treated is well water, nanofiltration also needs to be preceded by microfiltration to extend membrane service life.
In order to avoid depositions in the membranes during periods of inactivity, it is necessary to rinse the membranes with permeate water.

*Table 6. Removal of electrolytes from clean process water*

| Specific objective | | | |
|---|---|---|---|
| Removal of $Ca^{2+}$ from clean water used for steam generation in a cogeneration boiler. | | | |
| **Origin of the water** | | | |
| Groundwater extracted from a well. | | | |
| **Membrane used** | **Working conditions** | | |
| | **P (bar)** | **Permeability ($l/hm^2bar$)** | |
| NF-90 | 5 | 6.7 | |
| **Results of the removed parameters** | | | |
| **Parameter** | **$C^{Feed}$** | **$C^{Permeate}$** | **Retention (%)** |
| Conductivity (μS/cm) | 1414 | 34 | 98 |
| Chlorides (mg/L) | 55 | 2.2 | 96 |
| Sulphates (mg/L) | 240 | 1.1 | 100 |
| Sodium (mg/L) | 18 | 6.3 | 65 |
| Calcium (mg/L) | 71 | 1.3 | 98 |
| Magnesium (mg/L) | 16 | 0.4 | 98 |
| **Uses of the treated water** | | | |
| The water treated by nanofiltration could be subjected to a reverse osmosis process to further remove electrolytes and obtain purer water that would avoid scaling problems in the cogeneration boiler. The use of nanofiltration enables delcalcifiers to be replaced which, although possibly more effective in removing calcium, generate brines containing high concentrations of electrolytes with high conductivities, which are difficult to handle. The residue generated by nanofiltration had much lower electrolyte concentrations: these were even lower than those obtained by reverse osmosis and are therefore easier to handle.<br>The use of nanofiltration before reverse osmosis assures optimum nanofiltration efficiency and increases the service life of the osmosis membranes. | | | |
| **Observations** | | | |
| In this case as well, although the water to be treated is well water, nanofiltration needs to be preceded by microfiltration.<br>In order to avoid depositions in the membranes during periods of inactivity, it is necessary to rinse the membranes with permeate water. | | | |

*Tabla 7. Eliminación del calcio para sustituir un descalcificador*

| Specific objective |
|---|
| Removal of electrolytes from wastewater and from clean water used in spray-dried powder manufacture.<br>In this case, water was used that came from two different sources and consequently produced different results, which are detailed below. |

| Origin of the water | | | |
|---|---|---|---|
| Wastewater from cleaning operations in the same company and from other adjacent companies. | | | |
| Membrane used | Working conditions | | |
| NF-270 | P (bar) | | |
| | 5.5 | | |
| Results of the removed parameters | | | |
| Parameter | $C^{Feed}$ | $C^{Permeate}$ | Retention (%) |
| Conductivity (μS/cm) | 1467 | 505 | 66 |
| Chlorides (mg/L) | 99 | 64 | 35 |
| Sulphates (mg/L) | 182 | 4 | 98 |
| Sodium (mg/L) | 88 | 38 | 57 |
| Calcium (mg/L) | 126 | 19 | 85 |
| • Origin of the water | | | |
| • Clean groundwater supplied from a well. | | | |
| Membrane used | Working conditions | | |
| NF-270 | P (bar) | Permeability (l/hm²bar) | |
| | 5 | 11.6 | |
| Results of the removed parameters | | | |
| Parameter | $C^{Feed}$ | $C^{Permeate}$ | Retention (%) |
| Conductivity (μS/cm) | 927 | 477 | 48 |
| Chlorides (mg/L) | 73 | 69 | 5 |
| Sulphates (mg/L) | 211 | 18 | 91 |
| Sodium (mg/L) | 45 | 29 | 36 |
| Calcium (mg/L) | 73 | 24 | 67 |
| • Uses of the treated water | | | |
| The nanofiltered water could be used in preparing ceramic slurries. The removal of divalent inorganic salts caused deflocculant consumption to decrease, raising the slurry solids content and consequently considerably increasing spray dryer production with lower energy consumption. | | | |
| Observations | | | |
| Nanofiltration needs to be preceded by microfiltration to extend membrane service life. In the case of wastewater, physical and chemical treatment is also required. To avoid depositions in the membranes during periods of inactivity, the membranes must always be kept clean and disinfected with sodium bisulphite. | | | |

*Table 8. Removal of electrolytes from clean process water and from wastewater for use in the production process*

A further point of interest in nanofiltration processes, provided the technically required efficiencies are achieved, is the lower energy demand compared with that of competing techniques such as reverse osmosis, which operates at higher pressures with lower permeate flows and hence lower efficiencies, entailing higher treatment costs. When the costs of these treatments are quantified, the cost of NF is found to vary between 0.2 and 0.4 €/m$^3$, compared with at least 0.6 €/m$^3$ in the case of reverse osmosis.

## 5. CONCLUSIONS

The results compiled in this study allow the following conclusions to be drawn:

- Advanced filtration technologies, in particular nanofiltration ones, are techniques to be considered for achieving good, comprehensive water management in ceramic sector companies.

- Nanofiltration provides good results in removing organic compounds, boron, and different electrolytes present in the water. In addition, the data analysed in the study highlight differences in the performance of the nanofiltration membranes used. Thus, the NF-90 membrane was more effective in removing all the studied pollutants. However, the NF-270 membrane was more selective and provided good retentions at greater permeate flows than those achieved with the NF-90 membrane. Therefore, these performance features may be of interest for certain applications.

- Nanofiltration is a technology that enables the process to run at pressures that are not very high in comparison with those of similar technologies such as reverse osmosis, yielding similar results with higher treated-water output efficiencies. Consequently, the operating cost of nanofiltration and hence also the cost per treated cubic metre are lower than in the case of reverse osmosis.

## 6. ACKNOWLEDGEMENTS

This study was financially supported by the Valencian Institute for Small and Medium-sized Companies (IMPIVA) of the Valencia Region, the Centre for Industrial Technology Development (CDTI), the Eureka Programme, and the European Regional Development Fund (IMAETA/2005/11, IMCITA/2007/5, IMIDIE/2009/3, ∑!4121 and 38374/CDTI).

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