# Corrosion Studies using Zeolite Synthesized from Fly Ash

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# Abstract

**Background/Objectives:** Zeolite is emerging as an environmental-friendly alternative to hard chrome, cadmium and epoxy polymers based corrosion resistance coating for steel, magnesium and aluminium alloys. Owing to inert nature, low modulus of elasticity and high strength, zeolite coatings are being used as anti-corrosive coatings for metallic substrates. **Methods:** Molten Salt Method was used to convert fly ash to zeolite. The synthesized zeolite was characterized using X-Ray Diffraction (XRD) technique and Scanning Electron Microscopy (SEM). The synthesized zeolite was coated on mild steel samples using polymer binder (Polyvinylidene fluoride-PVDF). Zeolite-coated mild steel samples and uncoated samples were exposed to sea water for 2 months. The corrosion behaviour was found using Weight-loss method and Electrochemical Method (Potentiodynamic Polarization Method-Tafel Plot analysis). Corrosion studies of samples were also carried out in different acid solutions (Sulphuric Acid, Nitric Acid, Hydrochloric Acid) for a month. Corrosion rates of samples and inhibition efficiencies of coaed-samples were found using Weight-loss Method. **Findings:** Results showed that fly ash was converted into zeolite with Sodalite and Cancrinite as major crystalline phases. The optimum zeolite to binder ratio was found to be 1:0.3. The zeolite-coated samples had excellent corrosion inhibition efficiency in sea water (above 85%). They had corrosion inhibition efficiencies above 80% in acid solutions. **Application:** This study presents experimental results dealing with the possibility of transforming fly ash into a zeolite material and using the synthesized material as an effective anti-corrosive agent.

Keywords: Fly Ash, Zeolite, Weight-Loss Method, Tafel Plot, Corrosion Rate, Inhibition Efficiency

# 1. Introduction

The continuous reliability and safety of the public infrastructure and industries are necessary to the nation's quality of life, productivity in all sectors, economic competitiveness, security and defence. Every segment of the public infrastructure like railways, highways, waste treatment, energy management, water management, power generation, etc. is part of a complex system which involves large investment. Corrosion affects mostly all of the materials and structures used in infrastructure and the resulting losses every year is found to be in hundreds of millions of dollars. This makes corrosion as one of the most important problems in our modern societies. In August 2014, National Corrosion Council of India (NCCI) estimated the annual loss due to corrosion to be 4% of the Gross Domestic Product (GDP)<sup>1</sup>. In India, two lakh crore worth losses occur due to corrosion of steel every year. Worldwide 25,000 tons of steel is lost every minute due to corrosion<sup>2</sup>. Due to increase in the use of steel in infrastructure, the country is facing resourcecrunch and not in a position to bear huge losses on account of corrosion. The rapid industrialization of many countries indicates that the competition for and the price of metal resources will increase, which in turn will affect the econ-

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omy. Thus, our economy would be drastically changed if there was no corrosion<sup>3</sup>.

Conventionally, steel and aluminum alloys are protected by anticorrosive chromate coatings. But these coating contains carcinogenic hexavalent chromium ( $Cr^{6+}$ ) compounds. Sol-gel coatings of zirconium dioxide, silica dioxide, SiO<sub>2</sub>–TiO<sub>2</sub>, SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> and silanes are the other possible substitutions to chromate coating. But, inorganic coatings are susceptible to crack and these coatings do not provide acceptable protection against corrosion<sup>4</sup>.

While searching for environmental-friendly alternative to the traditionally used chromium and cadmium coatings, conventional wisdom has been focusing on materials with a high hardness that is usually provided by dense metals, along with high elastic modulus. A good anti-corrosive coating should have high hardness accompanied with relatively low modulus because high elastic modulus limits the coating's flexibility and thus limits its overall performance. Zeolite with its unique crystalline porous framework satisfies this condition. Zeolite's crystallinity offers high hardness and its porosity provides low elastic modulus which leads to excellent resilience and flexibility. So, zeolite is "greener" alternative to conventional corrosion resistance coatings<sup>5</sup>.

Zeolites are hydrated alumina silicates of the alkali and alkaline-earth metals with completely interlinked framework formed by corner sharing of silica ( $SiO_4$ ) and alumina ( $AlO_4$ ) tetrahedral. Zeolite has wide applications in waste water treatment and flue gas treatment. It is mostly used in catalysis, sorption and ion exchange<sup>6</sup>.

It has been illustrated in earlier researches that zeolite coating on steel and aluminum alloys provides a remarkable corrosion resistance7. The wear-resistant, anti-microbial properties8 and bio-corrosion control9 of these films have been confirmed. Ceramic coatings have shown ability to be used where corrosion and wear coexist, even at elevated temperature<sup>10</sup>. Investigations have shown the potential of using fly ash as raw material or various types of zeolite<sup>6-21</sup>. The inorganic residues obtained from combustion of coal are called Coal Combustion By-products (CCB) which consists of fly ash, slag, bottom ash and fluidized bed combustion and flue gas desulfurization by-products. Fly ashes are collected using electrostatic or mechanical precipitation of dust-like particles from flue gases arising from furnaces which use coal as fuel for combustion. The amount of fly ash generated by coal-based thermal power plant has been increasing at an alarming rate throughout the world and its disposal is a pressing issue<sup>11</sup>. Although a part of it is used by building industries, there is still a large amount which is disposed off in ponds and landfills<sup>12</sup>. Globally, more than 65% of fly ash produced from coal-based thermal power stations is disposed off in landfills and ash ponds and the costs required for land filling is very high. Fly ash has a broad range of hazardous effects on humans, animals, aquatic life and plants. Particulate pollution is one of the important reasons for the worsening of respiratory diseases like asthma, increasing the rate of premature mortality from heart and respiratory diseases in highly populated cities<sup>13</sup>.

As disposal of fly ash is difficult, its recycle has become an increasing concern these days for sustainable development. So fly ash is being utilised by different sectors of economy. As it has pozzolanic properties, it is widely used in cement industries, manufacturing of asbestos sheets, ready-made hollow blocks, road embankment and also used in agriculture<sup>12</sup>. Due to the similarity of the chemical and mineralogical composition of fly ash and other naturally occurring materials, many research studies regarding synthesis of zeolite from coal fly ash were initiated.

# 2. Materials and Methods

## 2.1 Materials

Zeolite was synthesized from coal fly ash using molten salt method. Fly ash was collected from North Chennai Thermal Power Plant. The chemicals used were of Analytical grade. Sodium Hydroxide (NaOH), Sodium Nitrate (NaNO<sub>3</sub>) and Tetra ethyl Ammonium Bromide were used as base, neutral and Structure Directing Agent (SDA) respectively. Zeolite was coated on mild steel. Binder used for coating was polymer-Polyvinylidine fluoride (PVDF) and the solvent used to dissolve the polymer is n-methyl 2-pyrolidone (NMP).

Table 1.Composition of fly ash

Elements	Weight percentage
SiO <sub>2</sub>	60.02
Al <sub>2</sub> O <sub>3</sub>	29.28
Fe <sub>2</sub> O <sub>3</sub>	4.93
CaO	1.6
MgO	0.5

## 2.2 Methodology

## 2.2.1 Synthesis of Zeolite (Molten Salt Method):

Mixture containing fly ash, NaOH and NaNO<sub>3</sub> in the ratio 0.7:0.3:1, along with the SDA, was ground to get fine powder form and was thermally treated at  $350^{\circ}$  C for 24 hours. The lump was cooled down to room temperature and then it was crushed and washed with deionised water to remove excess salts and base. Later the dried sample was taken for analysis.

#### 2.2.2 Sample Preparation and Zeolite coating:

Mild steel sheets were cut into size of  $2x1.5 \text{ cm}^2$  and polished with grit sand paper. The samples were washed using distilled water, acetone and acid (Diluted Hydrochloric acid). Zeolite was coated on both sides of samples manually. Zeolite to binder ratio was optimized and found to be 1:0.3. It was coated uniformly on cleaned mild steel samples and oven dried at around 80° C for 12 hours. The coating thickness was found using screw gauge and it was found to be 35µm on each side.

#### 2.2.3 Characterization Studies

X-ray Diffraction Analysis: The powder X-Ray Diffraction patterns of the zeolite samples were recorded on X- Ray Powder diffractometer,  $2\theta$  range of  $5-80^{\circ}$  and step size of 0.02. The X-Ray source was operated at  $30\mu$ A and 45kV. Samples for the XRD analysis were ground into a fine powder and about 0.25 to 0.5 g of sample were packed into sample holder and lightly compressed to make it tight and uniform. The XRD patterns of fly ash and synthesized zeolite were then recorded and analysed.

SEM Analysis: High-resolution images of surfaces of fly ash and synthesized zeolite were obtained using Scanning Electron Microscope (VEGA3 TESCAN). This type of microscope gives images of samples by scanning it using a focused beam of electrons. The interaction of electrons and atoms in the sample produces different signals which can be detected and it contains information about the sample's morphology.

## 2.2.4 Corrosion Measurement Techniques

Weight-Loss Method: In this technique, the sample metal of known weight and area is exposed to the corrosive environment for a particular period of time and the difference in weight before and after exposure is found out. Corrosion rate is commonly expressed in mils per year. Corrosion rates of samples are calculated using the following formula<sup>1</sup>,

Corrosion rate (mpy) = 
$$534w_1$$
/DAT (1)

where  $w_1$  - weight loss in mg T- time of exposure in hours A- surface area of sample in inch<sup>2</sup> D- density of sample in g/cm<sup>3</sup> mpy = mils/year (1 mil = 1 milli inch= 0.0254 mm)

Inhibition efficiency IE % =  $[(r-r_1)/r] \times 100\%$  (2)

where r and  $r_1$  are corrosion rates of uncoated-mild steel sample and zeolite-coated mild steel sample respectively.

Electrochemical Method: As most of the corrosion process in aqueous solution are electrochemical in nature, different electrochemical techniques have been employed for the determination of corrosion rates like Cyclic potentio dynamic polarization, Polarization resistance, Potentio-static test, Electrochemical Impedence Spectroscopy and Cyclic voltammetry. These corrosion measurement techniques are currently popular among corrosion engineers, primarily due to its rapidity. Conventional corrosion measurements like weight loss determinations take days or weeks to find the corrosion behavior, but an Electro chemical experiment will require few minutes and at most few hours.

The electrochemical analyser consists of three electrode system with working electrode (sample), standard electrode (Ag/AgCl) and auxiliary electrode (Platinum wire). This method measures the current in an electrochemical cell under conditions for the applied voltage. The potential was measured between the standard and working electrodes, and the current was measured between the working and auxiliary electrodes. Potential versus logarithm of absolute current (Tafel plot) generated from this experiment were used to investigate the corrosion behaviour of metals. These plots were recorded in the scan range of -1.1 to -0.2 V and the scan rate was 0.01V s<sup>-1</sup>.

# 3. Results and Discussions

## 3.1 Characterization Techniques

## 3.1.1 X-ray Diffraction Analysis

The XRD pattern of the raw fly ash (Figure 1) was analysed. It shows that the fly ash consists of Quartz (SiO<sub>2</sub>), Mullite  $(3Al_2O_3.SiO_2)$  as major crystalline phases with small amounts of Magnetite  $(Fe_3O_4)$  and Hematite  $(Fe_2O_3)$ . The most intense peak on the XRD pattern was found at 26.6° 2 $\theta$  which was identified as Quartz and the less intense peaks were Mullite, Hematite and Magnetite. Fly ash's XRD pattern also shows a broad hump in the region between 17 to 32° 2 $\theta$  indicating that the fly ash contains amorphous glassy phase<sup>13</sup>.

Alkaline Hydrothermal method is widely used for the synthesis of zeolite. The conventional method requires a high ratio of solvent to raw material that results in less productivity and so it becomes tedious to use it in massive synthesis of zeolite. And also large quantity of liquid waste is produced from this method of synthesis even after treatment. The synthesized zeolite consists of different phases, is less pure and structurally heterogeneous which limits the applications of the product. In this research work, zeolitic materials were synthesized from fly ash by the treating it thermally along with alkaline molten-salts which can overcome the disadvantages due to hydrothermal method.

On comparing the XRD pattern of the synthesized zeolite (Figure 2) and raw fly ash (Figure 1), vital changes were observed, such as, the disappearance of the broad hump between 17-  $32^{\circ} 2\theta$ , disappearance of Quartz, Mullite and Hematite peaks which confirms the dissolution of amorphous phases and crystalline phases which were actually seen in the XRD spectra of the raw fly ash. And the stabilisation of base line was also observed.

Initially amorphous components were utilized for zeolite formation, and later mullite and quartz were used

after the complete decomposition of amorphous components by the base used in the experiment. NaOH acts as an activator and attacks different forms of Aluminium and Silica components in the fly ash and make them available for zeolitisation. Na<sup>+</sup> cations plays a significant role in stabilizing the sub-building units of zeolite frameworks, the action of Na<sup>+</sup> cations in the molten conditions are similar to that in aqueous system<sup>16</sup>. NaOH can also act as fillers of zeolitic pores.

Reaction temperature in hydrothermal method of synthesis of zeolite influences crystallization kinetics and the resulting phases formed in the product. Generally, increase in temperature accelerates crystallization process to form zeoite<sup>17</sup> and high temperature synthesis forms dense zeolite phases, namely, Analcime, Cancrinite and Sodalite<sup>18</sup>. Here, under alkaline molten conditions, with NaOH as mineraliser and with help of SDA, fly ash was converted into zeolite with Sodalite and Cancrinite as major crystalline peaks after 24 hours treatment period.

## 3.1.2 Morphological Characterization by SEM

Figure 3 shows the morphological structures of raw fly ashes were obtained using Scanning Electron Microscope. The SEM image of fly ash shows that fly ash is heterogeneous in nature consisting of irregular spherical particles of various sizes, and it confirms the presence of Si and ashes which matches the literatures<sup>16-21</sup>. The size of particles varies from micro to nano. Figure 3 shows the raw fly ash was converted into zeolite of irregular spherical shape particles of varying size.



Figure 1. XRD of fly ash.



Figure 2. XRD pattern of Zeolite.



Figure 3. SEM images of (a) raw fly ash and (b) zeolite.

## 3.2 Corrosion Studies in Sea Water

Mild steel is an engineering structural material which is used abundantly in offshore and marine engineering, chemical processing, petroleum, refining industries, pipelines, mining and construction, etc. because it has good mechanical and physical properties, like plasticity, hardness, toughness, ductility, brittleness, welding and cutting performance, etc. and is the cheapest type of steel available. Corrosion reduces the life time of process equipment and pipelines which leads to leakage, loss of resources and products, environmental pollution, failure of entire structure and even loss of life. In cooling systems, storage tanks, and water transport pipelines for injection systems, etc., seawater is used which leads to corrosion problems, as corrosive natural electrolytes are present in large amount in seawater<sup>22</sup>. Thus corrosion is the most important cause for structural deterioration in marine and offshore structures. Table 2 shows the characteristics of sea water used in the experiment.

When mild steel is immersed in sea water, ferric oxide,  $Fe_2O_3$  (hematite) is formed when iron metal reduces water.

Ph	8.5
Electrical Conductivity	5.04S/m
Total Dissolved Solids(TDS)	35500ppm
Chemical Oxygen Demand(COD)	6240 mg/l
Salinity	35ppt

$$2Fe + 3H_2O = Fe_2O_3 + 3H_2$$
(3)  

$$4Fe + 3O_2 = 2Fe_2O_2$$
(4)

Hematite is also formed when steel is exposed to atmosphere but it is a very slow reaction. In pure water the first reaction (eq. 3) is also very slow. This is because there are actually two reactions taking place at separate sites. Steel (iron) was oxidized at one site and water was reduced to hydrogen at another site. The presence of salt in sea water forms an electrolytic solution that will conduct electricity. Electrolytic current flowed through the salt solution between the sites. Improved electrical conductivity of sea water leads to increased pitting corrosion which is regarded as the most hazardous form of corrosion. The corrosion behaviour of uncoated-mild steel samples and zeolite-coated samples were found using weight loss method and Tafel plot analysis.

#### 3.2.1 Weight Loss Methods

Figure 4 shows that the weight loss of coated sample was less when compared to the uncoated mild steel samples exposed to sea water during different time periods. Using equation 1 and 2, the corrosion rates and inhibition efficiency were found:

Corrosion Rate of coated sample  $(r_1) = 0.78$  mpy Corrosion Rate of uncoated sample (r) = 6.141 mpy

Inhibition efficiency of zeolite-coated mild steel was found to be 87.5%

Corrosion rate(mpy)	Resistance to Corrosion
Less than 1	Outstanding
1-5	Excellent
5-20	Good
20-50	Fair
Greater than 50	Not Suitable

**Table 3.** Relationship between corrosion rate andcorrosion resistance



**Figure 4.** Weights of Samples exposed to sea water during different time intervals.



**Figure 5.** (a) Zeolite- coated sample and (b) uncoated mild steel sample after exposing to sea water.

## 3.2.2 Tafel Plot Analysis

While conducting electrochemical experiment, the metal sample with a known surface area was used to know the corrosion behaviour of the metal in a corroding system. The metal sample along with counter and reference electrodes were immersed in a solution similar to the metal's environment (5% NaCl solution). Then the electrodes were connected to the potentiostat which allows to change the potential at a specific rate and measure the corresponding current that flows in the system.

Graphs were plotted between potential versus logarithm of current (Tafel plot). When a potential is applied to the metal undergoing corrosion in contact with the solution, a net current I flows through the metal. The curved line is the total current (I) which is the sum of the anodic current ( $I_a$ ) and cathodic current ( $I_c$ ). The sharp point in the curve is the point where the current reverses polarity as the reaction changes from anodic to cathodic, or vice versa. The sharp point is obtained by plotting potential against logarithmic of current. The onset of passivity in corrosion experiment leads to large changes in current, often it changes by six orders of magnitude. Thus logarithmic axis is used to record wide range of current values.

Tafel plot analysis was done by extrapolating the linear segments of a log of current versus potential graph back to their intersection. The intersect points of cathodic and anodic currents gives the corrosion current ( $I_{corr}$ ) and corrosion potential ( $E_{corr}$ )<sup>23,24</sup>.

The plots in figure 6 indicates that the coating modifies the mechanism of cathodic reaction and also anodic dissolution of iron, which indicates that the zeolite coating effectively protects the mild steel from corrosion. There was observable reduction of pitting corrosion due to zeolite coating<sup>5</sup>. The  $E_{corr}$  value of uncoated sample shifted to more negative value which indicates sample's resistance to corrosion had reduced after exposing to sea water<sup>25</sup>.

Corrosion rates of the mild steel samples in mpy were found using the following equation<sup>26</sup>

$$CR = 0.13I_{corr} EW/DA$$
 (5)

where  $I_{corr}$  – Corrosion current in  $\mu A$ EW – Equivalent weight in grams/equivalent A-Sample area in cm<sup>2</sup>

Tafel constants ( $\beta a$  and  $\beta c$ ) were calculated from anodic and cathodic portions of the Tafel plots. The corrosion parameters ( $I_{corr}$ ,  $\beta_a$ ,  $\beta_c$ , corrosion rate) obtained for blank, uncoated and coated mild steel samples are tabulated in Table 4. Corrosion current  $I_{corr}$  decreases in presence of

Table 4.Electrochemical testing results

Samples	I <sub>corr</sub> (μA)	$\beta_a$	β <sub>c</sub>	$\left  egin{array}{c} R_{_{p}} \ (\Omega) \end{array}  ight $	Corrosion rate (mpy)
Blank sample	640.2	12.719	2.739	44	4.56
Uncoated sample exposed to sea water	827.2	4.292	6.879	11	6.87
Coated sample exposed to sea water	30.33	19.710	3.393	615	0.43



Figure 6. Tafel plots of blank, coated and uncoated samples.

zeolite coating which indicates a change of the mechanism of cathodic and anodic process<sup>27</sup>. Corrosion rate is directly proportional to corrosion current, which clearly shows that the zeolite coating greatly hinders the mild steel corrosion.

It was noticed that the polarization resistance (Rp) of the coated sample increased in coated samples when compared to the uncoated sample. Both coating thickness and tight packing of zeolite contributed to the increase in polarization resistance and increased corrosion resistance<sup>4</sup>.

The inhibition efficiency of the zeolite-coated samples after exposing to sea water for 2 months was found using eq. 2:

IE =93.74%

#### 3.3 Corrosion Studies in Acid Solutions

Mild steel is the most commonly used metal which relatively inexpensive and possess metal properties that making it adequate for many uses especially in food, petroleum, chemical and electrochemical industries and power production. However, this metal undergoes deterioration where it is exposed to acidic medium like sulphuric acid and hydrochloric acid, which are normally used in industry for pickling and de-scaling of metals<sup>28</sup>. Mild steel is one of the main construction materials used in allied industries which handle acid, alkaline and salt solutions<sup>29</sup>. Intense care should be taken while selecting materials to handle the acids because acids are very corrosive to mostly all metals and alloys even when present in low concentrations in solutions.

When mild steel was dipped in test solution containing acid, bubbles were formed due to hydrogen evolution.



**Figure 7.** Weights of Samples in Sulphuric Acid Solution.



Figure 8. Weights of Samples in Nitric Acid Solution.



Figure 9. Weights of Samples in Hydrochloric Acid Solution.

The hydrogen evolution and dissolution of iron which leads to mass loss is due to the following reaction.

Fe (solid) + 2 H<sup>+</sup>
$$\rightarrow$$
 Fe <sup>2+</sup> + H<sub>2</sub> (gas) (6)

For example, when mild steel is dipped in solution containing Hydrochloric acid,

Fe (solid) + 2 HCl (aq) 
$$\rightarrow$$
 Fe Cl<sub>2</sub> (aq) + H<sub>2</sub> (gas) (7)

When coated samples were dipped in test solution, no bubbles were formed which means there was no contact between the metal and its environment, indicating that the coating is an effective barrier. The corrosion rate and inhibition efficiency of the coating were found using weight loss method.

Figure 7, 8 and 9 shows weights of uncoated mild steel samples and zeolite-coated mild steel samples exposed to diluted solutions of Sulphuric acid, Nitric acid and Hydrochloric acid respectively during different time intervals. It is clear from these figures that the weight loss of coated sample was less when compared to the uncoated mild steel samples exposed to different acid solutions over a period of time.



**Figure 10.** Zeolite-coated samples (a), (c), (e) and uncoated mild steel samples (b), (d), (f) after exposing to  $H_2SO_4$ , HNO<sub>3</sub> and HCl solutions respectively.

amples			
Samples	Corrosion	Corrosion rate	Inhibition
exposed	rate of coated	of uncoated	efficiency
to	samples(mpy)	samples(mpy)	

Table 5.

Corrosion rates of coated and uncoated

to	samples(mpy)	samples(mpy)	emerency
dil.H <sub>2</sub> SO <sub>4</sub>	0.79 (Outstanding Corrosion Resistance)	18.33	95.69%
dil.HCl	2.95(Excellent Corrosion Resistance)	30.86	90.44%
dil.HNO <sub>3</sub>	6.29 (Good Corrosion Resistance)	38.40	84.44%

Table 5 shows the corrosion rates and inhibition efficiencies of the samples in different acid solutions. The corrosion resistance of coating exposed to sulphuric acid solution was found to be very high when compared to other solutions<sup>30</sup>. Results showed that nitric acid was more corrosive than other acids. Nitric acid being a strong oxidizing agent, its corrosion effect on samples is very significant. Generally, an autocatalytic mechanism has been proposed to reason out the higher rate of corrosion in nitric acid solution. The primary displacement of H<sup>+</sup> ions from the solutions is followed by nitric acid reduction rather than hydrogen evolution because the reduction of acid leads to a notable decrease in free energy.

$$Fe + 4HNO_3 \rightarrow Fe(NO_3)_2 + 2H_2O + 2NO_2 - \dots$$
(8)

This reaction leads to evolution of nitrogen (II) oxide and formation of  $Fe(NO_3)_2$  which is the cause for further coloration of the medium<sup>31</sup>.

Results also showed that dissolution of metals was high during the initial days, and then eventually slows down due to formation of passivating corrosion complexes that protects the sample's surface from the corrosive environment. Therefore, table 5 shows that the zeolite-coated mild steel samples have better corrosion resistance than the uncoated samples.

# 4. Conclusion

Thus this project deals with utilization of waste for producing a valuable product, i. e., fly ash was converted into zeolite which is found to be an effective anti-corrosive agent. The synthesized zeolite coated on mild steel had excellent corrosion resistance in sea water and good corrosion resistance in various acid solutions. Zeolite-coated samples had excellent corrosion inhibition efficiency almost 96% in sulphuric acid solutions.

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