

# Effect of De-icing Chemicals on Strength and Microstructural Properties of Concrete

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**ABSTRACT-** Degradation of Concrete may occur through a variety of physical and chemical processes. This research deals with the chemical process in which cement paste and de-icing salts are involved. It is known fact that the use of de-icing salts causes corrosion of steel in reinforced concrete and leads to structural failures. However, the effect of de-icing salts on degradation of concrete is not fully researched.

Although application of de-icing salt is mandatory to maintain serviceability of concrete pavements in winter conditions, penetration of salt into concrete may be detrimental to both concrete and underlying reinforcement.

**KEYWORDS-** De-icing Chemicals, Micro Structural Study, Compressive Strength, X-ray Diffraction Analysis, Scanning Electron Microscopy (SEM).

## I. INTRODUCTION

### A. General

Concrete is one of the most widely used construction material in the world. It is composed of three components: cement, aggregates and water. Water being an essential component of concrete, combining water with a cementitious material forms a cement paste by the process of hydration of cement [1]. The cement paste bonds the aggregate together, fills voids within the concrete, and allows it to flow more freely. Less water in the cement paste will produce a stronger, more durable concrete, more the water more will be free-flowing concrete with a higher slump. Impure water used to make concrete can cause serious problems when setting of concrete or in causing premature failure of concrete structures. And it has been found that impurity in water samples used in mixing concrete can impair the strength of concrete especially the compressive strength of concrete [6]. In a similar way, water used for curing concrete can impair the strength of the concrete. Impurities and deleterious substances which are largely introduced from water used in mixing concrete are likely to interfere with the process of hydration, preventing effective bond between the aggregates and matrix. The impurities sometimes reduce the durability of the aggregate [1]. Excessive impurities in mixing water may affect not only setting time, concrete strength, and volume stability, but may also cause efflorescence. Where possible, water with high concentrations of dissolved solids should be avoided. Salts or other deleterious substances contributed

from the aggregate or admixtures are additive to those that might be contained in the mixing water. These additional amounts are to be considered in evaluating the acceptability of the total impurities that may be deleterious to concrete.

Concrete is very significant construction material used for bridges, buildings and many other structures. For the optimum working of concrete structures, the structures must be cured between temperature ranges of 10°C to 40°C. The performance of ordinary Portland cement concrete mainly depends on the properties of the binder in the cement and the conditions which the concrete might be exposed. The rate of hydration is one of the strong factors affecting the development of strength of concrete [7]. At low magnitude of temperature of hydration rate gets reduced, and setting time gets elongated and the strength development is decreased by 20%-40%. During freezing, when the temperature drops below -4 (degree), the moisture migrates within the matrix of binder and ice starts to hinder hydration process. When ice gets solidified it will expand up to 9 percent causing significant strains and stresses in concrete and on the pore walls of concrete.

In order to lessen the effects of low temperature causing stresses, various deicing chemicals are used. However these de-icing chemicals when react with various constituents of concrete lead to formation of certain compounds that may be detrimental to concrete, such as loss of compressive strength, tensile strength and durability[8].

In some places where temperature reaches below 0°C it will become difficult for the concrete structure to attain required strength when it is covered with snow like bridges, slabs etc. In order to keep the winter highways, bridges, slabs clear of ice and snow, de-icers are applied. De-icers mainly contain chlorides of salts containing mostly NaCl, MgCl<sub>2</sub>, CaCl<sub>2</sub>, UREA etc.[1]. The increasing usage of de-icing chemicals has raised concerns over their usage and are stated to have harmful effects on concrete structure through their reactions with the paste of cement and thereby reducing concrete integrity and strength. This study is aimed to investigate the effect of NaCl and UREA solutions on the compressive and micro structural properties of concrete. A total of 63 concrete cube specimens were casted and exposed to different concentrations of (NaCl and UREA) solutions as well as in plain water over a period of 3 months under natural winter conditions prevailing in kashmir. The effects of de-icing chemicals on concrete will be evaluated based on their compressive and micro

structural properties as compared to plain water cured concrete of similar age and condition [1].

## II. LITRATURE REVIEW

Korohonen C.J et al [3] introduce the antifreeze admixture for cold regions concreting using calcium chloride along parameters compressive strength resulting 2 - 4 % wt of 2 appeared to be most efficient up to -7c. Higher dosage leads to expansive oxychloride susceptible to reinforcement corrosion.

Kejin Wang et al. [4] proposed the damaging effects of de-icing chemicals on concrete materials. Using sodium chloride, calcium chloride with and without a corrosion inhibitor. Potassium acetate and an agricultural product along parameters mass loss compressive strength micro structure resulting paste immersed in NaCl and water showed steady mass gain while as paste immersed in other salts displayed a different degree of mass loss. Scaling paste immersed in water and NaCl showed no scaling while as paste immersed in cacl2 showed significant scaling.

Fatma Karagol et al. [5] explained the behavior of fresh and hardened concretes with antifreeze admixtures in deep freeze low temperatures and exteriors winter conditions using urea, calcium nitrate along parameters workability strength under test 4.5% urea 4.5% calcium nitrate resulting the average slump of control mixes was 4 cm. The average slump of the calcium nitrate, urea and the combination of calcium nitrate and urea mixes were 6, 12 and 22 cm respectively. Compressive strength best results were obtained for concrete at -5c and -10c showing strength of 41.91Mpa and 24.28Mpa respectively after 28 days.

Mastafa et al [5] explained about the effect of antifreeze use of physical and mechanical properties of concrete produced in cold weather ELSEVIER 27 FEB 2013 Using calcium nitrate and hydro xyethylamine mixture we used along parameters, The concrete was frozen in 0c, -5c, -10c, -15c, -20c for 2 days and then cured in normal water for 28 days. Resulting 30% calcium nitrate and 5% ethylamine gave the best performance.

## III. OBJECTIVES OF THE RESEARCH

After thoroughly studying the literature following are the objectives which are studied during this project:

- To study the effect of various concentrations of de-icing chemicals (NaCl and UREA) on hardened properties of concrete.
- To study the microstructure of concrete specimens cured using de-icing solutions.

## IV. SCOPE OF THE STUDY

This study will provide benefit to the concrete agencies which are there for concrete preservation, maintenance engineers, and many stakeholders who are interested in optimizing the ice and snow control activities without sacrificing concrete infrastructures. The main findings can help to guide the selection and implementation of the de-icing programs and thus make appropriate decisions for preventing concrete infrastructure from the impact of various de-icing salts [2].

The effect of urea on corrosion of reinforcement has been studied by few investigators only to a limited extent. The

research has generally been carried out on steel in contact with solutions of urea and not with concrete, and furthermore the range of solution conditions over which urea may be regarded as corrosive to steel has not been clearly established [2] [9].

## V. RESEARCH METHODOLOGY

### A. Materials

#### • Cement

A construction material that hardens, sets, and attaches to other materials to tie them together. Khyber Cement of 43 grade equivalent to Type I cement of ASTM C 150 was used as binder conforming to IS: 8112-1989. Various tests performed on cement and the results obtained are mentioned in Table 1 shows the physical properties of cement whereas chemical composition is shown in table 1 which is obtained performing quantitative phase analysis of X-ray fluorescence output of cement presented [10].

### B. Aggregates

Properties of aggregates govern the properties of mixture (concrete) in which it is used.

#### • Coarse Aggregates

Crushed stones up to 20mm in size are retained on 4.75mm I S sieves. Coarse Aggregates are used as filler in concrete to make it a more homogeneous mass. Crushed stone available locally were used as coarse aggregates whose physical properties are presented in table 3. The cleaned coarse aggregate is chosen and tested for various properties such as specific gravity, fineness modulus, bulk modulus etc. The physical characteristics are tested in accordance with IS: 2386 – 1963.

#### • Fine Aggregates

It is used to fill gaps between aggregates. By shaping the bulk, it reduces the cost of mortar or concrete. It avoids shrinkage and breaking of the material. It's easily accessible. The locally available river sand is used as fine aggregate in the present investigation. River sand with physical properties given in table 3 was used as fine aggregate. The cleaned fine aggregate is chosen and tested for various properties such as specific gravity, fineness modulus, bulk modulus etc. in accordance with IS: 2386-1963.

### C. Water

Water is an important constituent in concrete. It chemically reacts with cement (hydration) to produce the desired properties of concrete. Mixing water is the quantity of water that comes in contact with cement impacts slump of concrete and is used to determine the water to cement ratio (w/c) of the concrete mixture. Strength and durability of concrete is controlled to a large extent by its water cement ratio. Mixing water in concrete includes batch water measured and added to the mixer at the batch plant, ice, and free moisture on aggregates, water included in any significant quantity with chemical admixtures, and water added after batching during delivery or at the jobsite. Water absorbed by aggregates is excluded from mixing water. Besides its quantity, the quality of mixing water used in concrete has important effects on fresh concrete properties, such as setting time and workability; it also has important effects on the strength and durability of hardened concrete [4].

In general, the quality of water used in construction is the same as that used in drinking water. This is done to ensure that the water is relatively free of impurities such as suspended solids, organic matter, and dissolved salts, which can have an adverse effect on the properties of the concrete, particularly the setting, hardening, strength, durability, and so on. The pH value of water used for concrete construction must be in the range of 6-7.5. The water must be clean and free of sugar, molasses, or their derivatives, as well as sewage, oils, and organic substances. If the quality of the water to be used for mixing is in doubt, make 75 mm cubes in cement mortar 1:3 mix with distilled water and with the water in question separately. The latter type of cubes should achieve 90% of the strength obtained in cubes with the same amount of distilled water after 7 days. Alternatively, the water must be tested in an approved laboratory before being used in the preparation of concrete/mortar. Water quality for construction must be tested or monitored on a regular basis because it affects the overall strength of the concrete [4]. The following are the permissible solids limits for plain and reinforced cement concrete:

Table 1: Permissible limits of impurities in plain and reinforced concrete

Types of solid in water	Permissible limits
Organic matter	200mg/l
Inorganic matter	3000mg/l
Sulphates (SO <sub>4</sub> )	400mg/l
Chlorides(Cl)	2000mg/l (PCC) and 500mg/l(RCC)
Suspended matter	2000mg/l

• **Function of Water during the Production of Concrete**

Water wets the surface of aggregates, facilitating the spreading of cement over the aggregates and makes the mix workable, initiates the hydration process of cement subsequently starts the setting and hardening process and controls the heat generation which is the biggest concern of mass concrete structures by hydration process of the cement [4].

**D. Superplasticizer (Sps)**

Super plasticizers are same as that of plasticizers in terms of their action but are chemically different from plasticizers. The effect of plasticizer is comparatively more than that of plasticizer such that for the same dose they can permit the reduction of water content by up to 20%. Superplasticizers (SPs), also known as high range water reducers, are additives used in making high strength concrete. Plasticizers are chemical compounds that enable the production of concrete with approximately 15% less water content. Super plasticizers allow reduction in water content by 30% or more. These additives are employed at the level of a few weight percent generally added in the amount of 0.1 to 4% by weight of binder. Plasticizers and super plasticizers retard the curing of concrete. The super plasticizer used in this study is MasterPel777 by BASF @ 1% of binder throughout the study. One of its benefits is

that it can improve both early and final strength. In addition, slump retention and workability of concrete also enhanced by using MasterPel777 by BASF if compared with traditional super plasticizer. The table 2 is the descriptions on properties of the super plasticizer as supplied by the manufacturer.

Table 2: Properties of Super Plasticizer (Source: Manufacturer)

Physical state	Liquid
Color	Dark Brown
Freezing point	-5.0°C
Boiling point	>100°C
Chloride content	<0.2%
Relative density	1.035±0.02at 25°C
Reduction in permeability to water specimen	<50% of control mortar
pH value	≥6

**E. De-Icing Chemicals**

Various de-icing salts have been use on the roads; [11, 5] some of them are shown in the table 3

Table 3: Commonly Used De-Icing Chemicals [5]

Material Type	Snow and Ice Control Material	Primary Components
Chloride Salts	Sodium Chloride (NaCl)	Na, Cl
	Calcium Chloride (CaCl <sub>2</sub> )	Ca, Cl
	Magnesium Chloride (MgCl <sub>2</sub> )	Mg, Cl
Organic Products	Calcium Magnesium Acetate (CMA)	Ca, Mg, C <sub>2</sub> H <sub>3</sub> O <sub>2</sub>
	Potassium Acetate (KA)	K, C <sub>2</sub> H <sub>3</sub> O <sub>2</sub>
	Agricultural By-Products	Complex sugars; cheese brine
	Manufactured Organic Materials	Varies with product (i.e. glycol, methanol)
	Beet Juice	Beet juice mixed with salt brine
Nitrogen Products	Urea	Urea, Ammonia
Abrasives	Abrasives	Varies with the source of the material

In our study, we used urea and NaCl as de-icing salt as they are easily available in the market at affordable price; moreover NaCl has been used as a de-icing salt from very long time.

**F. Urea**

One of the most popular products used to melt ice (besides salt), it has many advantages. Urea is a fertilizer made from carbon dioxide and ammonia (not urine!) containing 46% nitrogen, and it is used to melt ice quite effectively. As compared to NaCl, it can be a better choice due to its less corrosive properties, meaning that metals, concrete, fabrics, and skin are not as irritated or damaged as they are by salt. This makes urea and good choice for people who want to avoid salt-related damage to their vehicles or homes and surfaces as shown in figure 1 [12].



Figure1: UREA

**G. Nacl**

Sodium chloride is most used de-icing salt in the world. Its easy availability, cost effectiveness and good salt melting properties make it one of the most used de-icing salts. The major problem is this salt is that it corrodes the reinforcement [13].

**H. Design Mix**

Design mix of M30 grade was prepared with cement content with proportion of various materials as under  
 Cement 400kg/m<sup>3</sup>  
 Fine aggregate 654 kg/m<sup>3</sup>  
 Coarse aggregate 1184 kg/m<sup>3</sup>  
 Water cement ratio= 0.43  
 Design mix was prepared using codes IS 456:2000 and IS 10262:1982

**VI. RESULTS AND DISCUSSIONS**

**A. Compression Test**

The most important property of hardened concrete is its compressive strength. Concrete compressive strength was measured at 28 days using standard cube specimens of 150mm150mm150mm. The compressive strength of concrete was tested using a Compression Testing Machine (CTM) with a capacity of 3000 kN. The concrete samples to be tested were brought out of the respective curing tanks few hours before they could be tested and air dried. The specimen was placed centrally between the bearing plates of the CTM, and a load of 140 kg/cm<sup>2</sup> /min was applied continuously and uniformly [1].



Figure 2: Compression testing machine with maximum capacity of 3000 Kn

The load was increased until the specimen broke, and the maximum load that each specimen could withstand was recorded. Compressive strength was measured three times for each sample and the compressive strength results were expressed as the average of these values as shown in figure 2.

**B. Compressive Strength Test Result**

Table 4: Compressive Strength Test Results of NaCl at Different Ages

	AGE	14 Days	28 Days	3 Months
1	Plain Water	29	32	43
2	3%(NaCl)	31.2	33	41
3	6%(NaCl)	30	33	38
4	9%(NaCl)	31.5	34	37.5

The early strength (compressive strength) of cubes at 28 days exposed to de-icing solution of NaCl gets increased a bit this is due to reason that NaCl has hydration accelerating properties , however the long term strength decreased with increase in percentage of salt.

Table 5: Compressive Strength Test Results of Urea((NH<sub>2</sub>)<sub>2</sub>CO) at Different Ages

S.No	Age	14Days	28Days	3Months
1	Plain Water	29	32	43
2	3% UREA	29.5	34	43.5
3	6% UREA	28	30	41
4	9% UREA	26	31	38

The early (28 days) strength of cubes exposed to urea solution remains as compared to control cubes . However the strength of cubes exposed to 3% urea solution remains same as control specimens and cubes exposed to 6% and 9% gets decreased as compared to control samples. The long term compressive strength of cubes exposed to urea solution gets decreased for 6% and 9% but remains almost same for cubes exposed to 3% as that of control specimen.

**C. Comparison Of Compressive Strength Test Results**

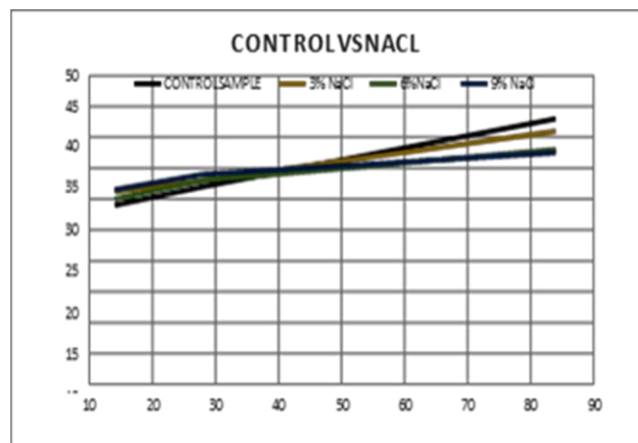


Figure 3: Comparison of test results of cubes Cured with plain water and different concentration of NaCl

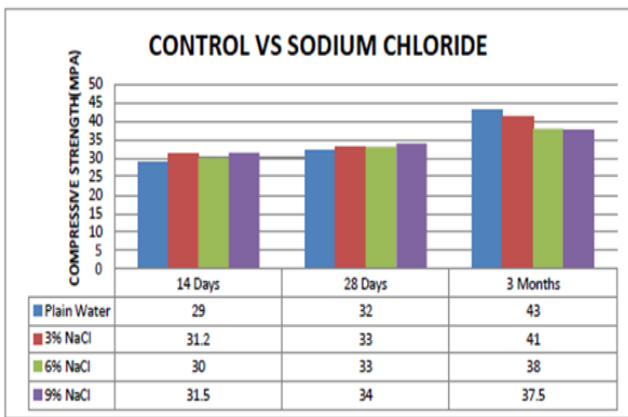


Figure 4: Bar Chart of Compressive Strength Results of NaCl Vs Control

The effect of NaCl as de-icer on early strength is not much significant and at the later stage the strength decrease, this is due to the formation of oxychlorides (which are expansive in nature) formed by consuming hydration products and chloride ions that are provided by NaCl (Botsman et.al)[14] term strength loss may also be attributed to formation of  $\text{Na}(\text{CO})_3$  which is found to be detrimental to concrete as shown in figure 3 and figure 4 (Reddy et.al)[15]

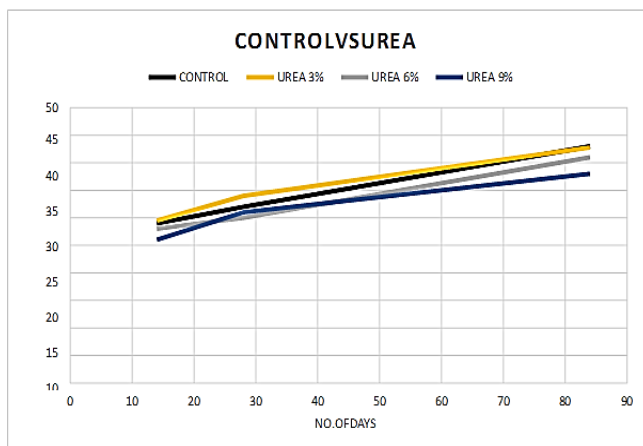


Figure 5: Comparison of Test Results of Cubes Cured with Plain Water and Different Concentrations of Urea

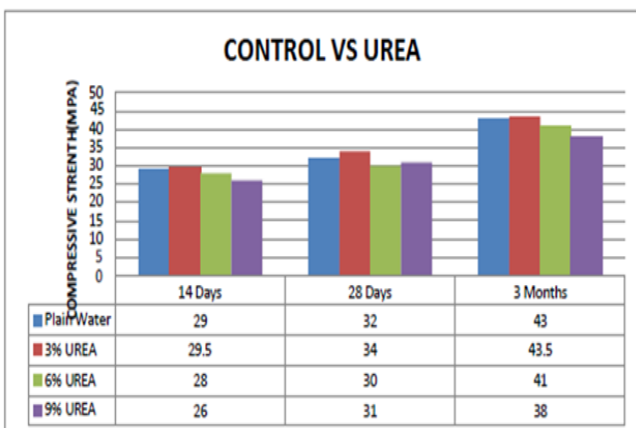


Figure 6: Bar Chart of Compressive Strength Results of Urea Vs. Control

As seen in the figure 5 and figure 6 of urea vs. control, the compressive strength at earlier stages of cubes remains same for control, 3%, 6% and 9% and decreases at later stages for 6% and 9% and remains same for control and 3%, this is due to the formation of ammonium carbonate and ammonium bi-carbonate. It has been found that ammonium bi-carbonate is more aggressive to concrete as compared to ammonium carbonate. This may be the reason for decrease of strength for 6% and 9%. For 3% urea also increases the solubility of C3S and C2S thereby increase the strength at 3%. However at higher percentages ammonium bicarbonate reason dominates the solubilisation of C3S and C2S reason.

**D. Microstructural Analysis**

The type, size, shape, quantity and dissemination of various phases present in concrete constitute its structure. The structure is dynamic in nature, starting from nanometer scale, to Micrometer scale, to millimeter scale. Study of Micro structural of concrete is a distinctive method to identify the morphological characteristics of concrete. X-Ray Diffraction Analysis (XRD) and Scanning Electron Microscope (SEM) are the universally adopted technique or method used to portray the micro structural compartment of concrete through hydration course.

X-Ray Diffraction Analysis (XRD)  
Scanning Electron Microscopy (SEM)

**E. X-Ray Diffraction Analysis (Xrd)**

The X-ray diffraction (XRD) method comprises of a suitable method to find the mineralogical investigation of crystalline solids. If a crystal-like mineral is brought in contact to X-rays of a specific wavelength then covers of atoms deflect the rays and create an array of peaks, which is representation of the mineral. The horizontal scale (diffraction angle) of a usual XRD pattern provides the crystal lattice spacing, and the vertical scale (peak height) provides the intensity of the diffracted ray. When the sample being X-rayed comprises of more than single mineral, the concentration of specific peaks from the specific minerals are proportionate to their amount [12].

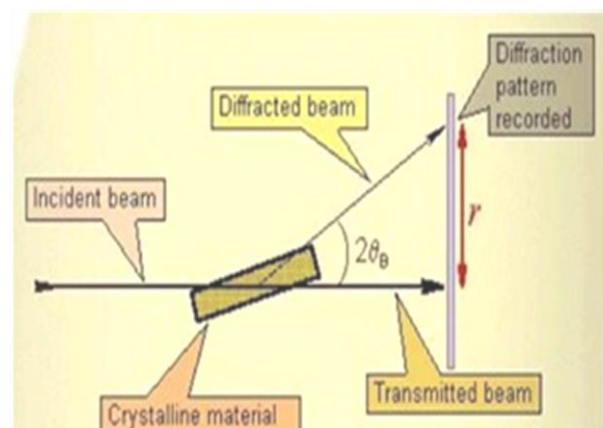


Figure 7: Schematic Diagram of X-Ray Diffraction

The XRD analysis is normally carried out at room temperature by means of powder X – Ray diffraction with filtered 0.154 nm Cu, K $\alpha$  radiation. Samples are scanned in a continuous mode from 100-800 with a scanning rate of 20 per minute. The X-Ray powder diffraction provides the graph between the X – Ray light intensity, which is

distributed on the sample and angle variance of the deflected X – Rays. The XRD method can be adopted to recognize the single crystals, and to make known the single crystal structure. It can be also adopted to recognize various crystals which are available in a mix, e.g. minerals in a stone. For minerals having inconstant formulations and structures (clays), XRD is the best technique for recognizing the formulations and finding their percentage in a sample.

**F. Scanning electron microscope (SEM):**

A scanning electron microscope is that kind of electron microscope which prepares images of a sample by scanning the top surface with a focused beam of electrons. A scanning electron microscope (SEM) scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition.

**G. SEM Image of NACL**

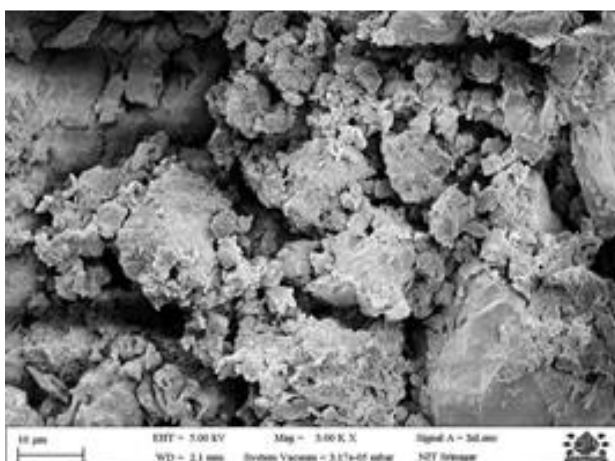


Figure 8: SEM IMAGE of 3% NACL

**Discussion on Results**

- The early strength (compressive strength) of cubes at 28 days exposed to de-icing solution of NaCl gets increased a bit thick is due to reason that NaCl has hydration accelerating properties , however the long term strength decreased with increase in percentage of salt.
- This may be due to the formation of calcium oxychloride, which is expansive in nature and is formed by reaction b/w chloride ions and calcium hydroxide (which is hydration product of cement).  
 $Cl + Ca(OH)_2 + H_2O = Ca(OH)_2 \cdot CaCl_2 \cdot 12H_2O$
- The long-term loss may be also attributed to the formation of various compounds which are formed by reaction between NaCl and hydration products of concrete, following compounds were formed:
  - SODIUM CARBONATE (Na<sub>2</sub>CO<sub>3</sub>)  
Sodium carbonate has been found to be decrease the strength of concrete (Reddy et.al)[15].
  - SODIUM ALUMINOSILICATE(Na(AlSi<sub>3</sub>O<sub>8</sub>))
  - SODIUM CALCIUM ALUMINIUM SILICATE HYDRATE(Na-CASH)  
 $(Na_3Ca_{3.36}Al_{19.48}Si_{26.52}O_{72}) \cdot (H_2O)_{24.48}$   
 CSH+ Aluminum + Sodium = Na-CASH  
 SODIUM CHLORIDE ACETATE(CH<sub>2</sub>CO<sub>2</sub>NaCl)

- The presence of these compounds was confirmed by xrd analysis. The other three compounds found in the NACL exposed cubes need to be researched again, to confirm their role in the decrease of strength. Results suggest that de-icers have strength accelerating properties at initial ages which discontinue at later ages. Also, the strength development patterns indicate that due to the presence of chloride environment, concrete specimens experience long-term compressive strength losses.
- SEM studies also suggest that at later ages the pore size gets increased which may be the reason for decreasing strength. Chlorides are also recognized to promote the leaching of Ca(OH)<sub>2</sub> and promote the development of porous CSH including complex reactions.
- Effect of urea on cubes: the early (28 days) strength of cubes exposed to urea solution remains as compared to control cubes . However the strength of cubes exposed to 3% urea solution remains same as control specimens and cubes exposed to 6% and 9% gets decreased as compared to control samples.
- The long term compressive strength of cubes exposed to urea solution gets decreased for 6% and 9% but remains almost same for cubes exposed to 3% as that of control specimen.
- The main product from this reaction which is carbonic acid (H<sub>2</sub>CO<sub>3</sub>) reacts with calcium hydroxide which is an important ingredient affecting hydration reaction and overall durability of concrete. Due to that, heat of hydration is reduced, shrinkage strain and carbonation resistance are greatly improved [16] but compressive strength is reduced at later stages for the cubes which are exposed to 6% and 9% urea solution.

**VII. CONCLUSION**

In this research it has been found that urea is a good de-icing agent as compared to NaCl, as urea causes least or no damage to the concrete at lower concentrations, however with increase in percentage of urea, it is found to be detrimental to the concrete. At an optimum concentration of 3% urea is most effective as a de-icing agent. The salt mostly used as de-icing salt is sodium chloride, which is found to cause damage to reinforcing bars as well as concrete. In this present study NaCl is also found to be accelerating the hydration at lower ages however long term strength is highly affected.

**VIII. RECOMMENDATION FOR FURTHER STUDIES**

- The current study was focused mainly on compressive strength of concrete cured in de-icers under prevailing winter conditions. However more study need to be one with regard to these de icers, some of which is enlisted under-
- Studies should be carried out for long term (1 YEAR AND ABOVE) compressive strength test for further comparison among the concretes exposed to different concentration of de-icing chemicals in water.
  - Studies should be carried out for the durability of reinforced concrete structures.
  - Study need to be done on the effect of these de-icers on other properties of concrete which include mass loss, water absorption, scaling etc.

- Studies should be carried out for different types and properties of cement, fine and coarse aggregates.
  - Studies should be carried out for large amount of NaCl or UREA in water.
  - Studies should be carried out for concrete cubes exposed to combination of various de-icing salts.
  - The effect of these de-icing chemicals on environment need to be studied.
  - The effect of these de-icers on reinforcement need to be studied especially UREA.
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### CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest between them and with any third party.

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