



## Enhancement of Corrosion Resistance in Fly Ash Blended Cement Concrete using Organic Inhibitors

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(Received: 31 March 2011;

Accepted: 24 August 2011)

AJC-10317

This paper presents the results of experimental investigations that were carried out to study the strength, micro structural and corrosion resistance characteristics of 30 % fly ash blended cement concrete with 3 % addition of organic inhibitors namely diethanolamine, benzimidazole, triethanolamine, triethylamine as corrosion inhibiting admixtures. The specimens were tested for compressive strength, split tensile strength, flexural strength, bond strength and water absorption test to ascertain their mechanical characteristics. The resistance to corrosion is evaluated based on the performance of the concrete for the penetration of chloride ions by means of impressed current technique. The possibilities of removal of chloride electrochemically and studies on migration of inhibitor were established. From the results obtained, it is found that the fly ash blended cement concrete increases the strength, reduces the permeability by the pozzolonic reaction and increases the durability of concrete with the addition of inhibitors. It offers good resistance against chemical attack and increases corrosion resistance in addition to increase in overall properties of concrete.

**Key Words:** Fly ash, Inhibitor, Chloride ingress, Corrosion resistance, Durability.

### INTRODUCTION

Concrete is one of the most widely accepted materials for civil engineering structures due to its inherent properties, especially its strength to sustain imposed loading condition. Corrosion of steel bars in the concrete has become one of the menacing problems on durability aspects which influence the service life of structures. Corrosion can be defined as the loss of useful properties of a material as a result of chemical or electrochemical reaction with its environment<sup>1,2</sup>. Corrosion is a well understood electrochemical phenomenon that takes place when the passivity is destroyed due to the carbonation of concrete in contact with steel or penetration of soluble chlorides right up to the reinforcement due to permeability or cracks<sup>3</sup>. The strong alkaline nature (pH of about 13) of Ca(OH)<sub>2</sub> prevents the corrosion of steel reinforcement by the formation of thin protective film on the metal surface passivity. The long-term durability of this protection against corrosion is connected with stability of conditions necessary for the passive layer. The performance of concrete to protect re-bar can be enhanced by various methods<sup>4,5</sup>. Among various remedies, use of supplementary cementitious material such as fly ash in concrete is an economical and efficient method to control corrosion<sup>6-8</sup>.

Selection of appropriate cementitious material is a critical factor for ensuring durability of concrete in severe exposure conditions. Fly ash, a by-product obtained from the combustion of coal in the thermal plants is removed by the dust collection system as fine particle residue from the combustion gases before they are discharged into atmosphere. Fly ash blended cements have received considerable attention due to their significantly better durability performance in severe exposure conditions<sup>9,10</sup>. Fly ash is an inexpensive replacement for portland cement used in concrete, while it actually improves strength, durability and ease of pumping of the concrete<sup>11,12</sup>. The use of fly ash in concrete as partial replacement of cement appears to constitute a very satisfactory outlet for this industrial by-product. In addition to the cost saving by partial replacement of cement, it also reduces CO<sub>2</sub> emission during the manufacture of portland cement<sup>13</sup>.

Corrosion resistant coatings in reinforcing bar, cathodic protection and addition of inhibitors are some of the methods to control corrosion. These inhibitors function by reinforcing the passive layer or by forming oxide layer and preventing chloride ions reacting from it<sup>14</sup>. This normally ranges from 10 to 15 mL/m<sup>3</sup> of concrete<sup>15,16</sup>. Therefore the corrosion protection characteristics of fly ash blended cement concrete can be further

improved by the addition of corrosion inhibiting admixture to the concrete mix<sup>17,18</sup>. To further elucidate its characteristics the permeability of concrete, chloride ion diffusion can be studied to evaluate its performance and the electro chemical removal of chloride technique can be adopted to enhance the corrosion resistance. Electrochemical chloride extraction (ECE) is essentially a simple process whereby chloride ions are removed from chloride contaminated concrete through ion migration. Many studies have been carried out on the removal of chloride in concrete and it was found that about 67 % of chloride could be removed by applying a current density of 1.0 A/m<sup>2</sup> for 200 h continuously<sup>19</sup>. It was also proved that the strength properties are not affected by the removal of chloride<sup>20</sup>. Numerous studies were carried out to determine the optimum percentage of addition of fly ash to replace cement. Study of literatures indicated that compared with the control mix, the concrete with 30 to 50 % cement replacement with fly ash show substantial increase in compressive strength, split tensile strength and flexural strength. Further, the data indicated that the corrosion rate of reinforcing steel in plain concrete specimens were about 13 to 19 times the corrosion rate of those in 30 % replaced fly ash concrete<sup>21,22</sup>. This paper is intended on the study of corrosion resisting inhibitors, which are added during casting of concrete in fly ash blended concrete in which cement is replaced by 30 % of fly ash.

## EXPERIMENTAL

Ordinary portland cement (43 grade) was used throughout the investigation and river sand with a fineness modulus of 2.06 conforming to grading zone III of IS 383-1970 was used as fine aggregate. Locally available well-graded granite aggregates of nominal size greater than 4.75 mm and less than 16 mm having a fineness modulus of 2.72 was used as coarse aggregates. Potable water has been used for casting concrete specimens. The water is free from oils, acids and alkalis and has a water-soluble chloride content of 140 mg/L. As per IS 456-2000, the permissible limit for chloride is 500 mg/L for reinforced concrete. High yield strength cold twisted deformed bar of Fe 415 grade conforming to IS 1786 has been used. Its mechanical properties are: yield strength of 475 N/mm<sup>2</sup>, ultimate tensile strength of 582 N/mm<sup>2</sup> and percentage of elongation on 30 cm gauge length is 11 %. Through experimental investigations the initial, final setting time of fly ash blended cement with inhibitor and without inhibitor have been determined and found to be satisfying the Indian standard codal specifications. Fly ash was obtained from Mettur Thermal Power Plant, Tamilnadu. In this study, 30 % by weight of cement is replaced by this class-C fly ash. In this experiment the grade of concrete adopted is M<sub>20</sub> whose mix proportion is designed as per I.S. method and arrived to be 1:1.464 : 3.21 with a water cement ratio of 0.50.

**Corrosion inhibiting admixture:** Organic inhibitors namely diethanolamine (DEA), triethylamine (TE), triethanolamine (TEA) and benzimidazole (BEZ) were used in this study. The percentage of each inhibitor added was 3 % by weight of cement. Chemical properties of inhibitors are given in Table-1.

TABLE-1  
PROPERTIES OF INHIBITORS

Inhibitor	m.p. (°C)	b.p. (°C)	m.w.	m.f.
DEA	154–155	270	105.10	C <sub>4</sub> H <sub>11</sub> NO <sub>3</sub>
TE	114.75	89.4	101.19	C <sub>6</sub> H <sub>15</sub> N
TEA	216	335.4	149.19	C <sub>6</sub> H <sub>15</sub> NO <sub>3</sub>
BEZ	171–173	–	118.14	C <sub>7</sub> H <sub>6</sub> N <sub>2</sub>

Experiments were conducted on concrete specimens to study their strength and micro structural properties. Strength tests include compressive strength, split tensile strength, flexural strength and bond strength. Determination of micro structural properties like percentage of water absorption, percentage of permeable voids and bulk density were obtained. These test results were compared with specimens cast with different corrosion inhibitors. Corrosion resistance was analyzed based on the following aspects: Studies on migration of chloride and inhibitor using diffusion test cell, electrochemical removal of chloride in concrete and injection of inhibitor into concrete by impressed current.

**Strength tests:** Concrete cubes of size 150 mm × 150 mm × 150 mm, beams of length 500 mm and cross section 100 × 100 mm, cylinders of size 150 mm diameter and 300 mm long were cast with and without inhibitors for determining compressive, flexural and split tensile strengths. After 24 h of casting, the specimens were demoulded and subjected to water curing. After 3, 7 and 28 days of curing, the specimens were tested as per IS: 516-1964. Cylinders of size 150 mm diameter and 300 mm long with rods of 70 cm length kept at the centre were cast and used for the determination of bond strength. Nine specimens were cast for each test and three specimens were tested at a time and the average values have been considered for comparison. Concrete cubes of size 150 mm × 150 mm × 150 mm were cast to determine the percentage of water absorption, percentage of permeable voids and bulk density. The tests were carried out as per ASTM C 642-97.

**Preparation of test specimen for diffusion test:** Eight numbers of concrete disks each of 85 mm diameter and 15 mm thickness were cast in a suitable mould. After 24 h, concrete disks were demoulded and cured for 28 days. After curing is over, the outer surface of the disk is covered with araldite to prevent evaporation of electrolyte solution.

**Assembly of cell:** The diffusion cell consists of anode, cathode with concrete disk and electrolyte. Each disk is fixed between PVC pipes of 85 mm diameter and 100 mm long on either side. Using fibre plastic square sheets of size 150 mm × 150 mm the two ends of the PVC pipes were closed. The concrete disk was fixed firmly between the two PVC pipes and M-seal is used to make the cell watertight. Thus concrete disk separates the cell into anodic compartments and cathodic compartments. In each compartment, titanium substantial insoluble anode of size 85 mm diameter was placed. This whole assembly is referred as diffusion test cell.

**Chloride analysis:** The cathodic compartment of the cell contains calcium hydroxide [Ca(OH)<sub>2</sub>] and sodium chloride [NaCl]. The anodic compartment of the cell consists of calcium hydroxide and an inhibitor. These inhibitors are added consequently with 100 and 200mm concentration. Electric current is applied to the cell at the rate of 1 A/m<sup>2</sup> through the electrodes.

Due to electrolysis process, sodium chloride was split into sodium ions and chloride ions.



Chloride ions are negatively charged which move through the disk to the opposite compartment (anodic compartment). The inhibitors are positively charged which move from anodic compartment to the cathodic compartment through the concrete disk. At the end of each 24 h, chloride analysis is done by taking 10 mL of solution from the anodic compartment using pipette. The procedure of chloride analysis is as follows. 10 mL of the pipette solution is taken in a conical flask. Adding two drops of phenolphthalein indicator, the solution turns pink indicating to alkaline nature. To neutralize alkalinity of the solution, it is titrated with 0.1 N sulphuric acid, which leads to disappearance of pink colour. Adding 3 drops of potassium chromate as indicator, the solution turns yellow. This solution is then titrated with 0.1 N silver nitrate solution, till the yellow colour turns to brick red. The corresponding burette reading is noted, from which chloride content can be calculated in ppm using following expression.

Chloride content = (volume of  $\text{AgNO}_3$ /volume of sample)  $\times$  normality of  $\text{AgNO}_3$   $\times$  molecular weight of chloride  $\times$  1000

Corrosion rate is determined to ensure the migration of inhibitors through the concrete disk. The weight loss rod for which the initial weight was already noted is inserted into the cathodic compartment of diffusion cell after 3 days of commencement of experiment. At the end of 12 days of the commencement of experiment, weight loss rod is removed and is put in a pickling solution for 15 min after, which it is cleaned and dried. Its final weight  $W_2$  is noted. The corrosion rate can be calculated by using the following formula.

Corrosion rate (mmpy) =  $(W_1 - W_2) \times 365 / (\text{DAT})$  where, D is the density of steel in  $\text{gm/mm}^3$ , A is the area of specimen in  $\text{mm}^2$  and T is exposure period in days.

### Electrochemical removal of chloride and injection of inhibitor into concrete by impressed current

**Preparation of test specimen:** The migration test proves the feasibility of migrating chlorides and inhibitor through concrete. Two RC slabs of 600 mm  $\times$  600 mm with a thickness of 85 mm were cast using M20 concrete. Each slab was provided with isotropic reinforcements of 12 mm diameter at 150 mm c/c. 8 mm rebar was connected to the mesh and this was projected outside to give electrical connection to rebar. 3 % of sodium chloride by weight of fly ash blended cement was added to the concrete at the time of mixing of concrete. Four numbers of weight loss rods of size 8 mm diameter and 100 mm long were tied with the mesh at four different locations. A bund was raised on all four sides of square slabs for a height and width of 50 mm using cement mortar 1:1. The RC slabs were cured for 28 days before conducting test.

**Assembly of system:** An anode mesh made of titanium substantial insoluble anode of size 150 mm  $\times$  50 mm was placed on top of the RC slab. A solution made of calcium hydroxide and inhibitor was poured on the slab. 1 A/m<sup>2</sup> of current is applied through the anode (titanium mesh) and cathode (rebar). Due to electrolysis process, chloride ions migrated towards the anode *i.e.* to the top of the slab, whereas positively charged inhibitor ions were migrated towards the rebar

(cathode) embedded in concrete. The current was applied for a period of 8 days. In case of untreated slab only the solution was ponded and no current was applied.

**Chloride analysis:** At the end of each 24 h chloride analysis was done using the procedure explained before. To know the efficiency of inhibitor injection in simultaneous with chloride removal the potential of the rebar was measured after 15 days of treatment. Both in treated and untreated slab the potential was measured over period of 60 days. The potential was measured at 10 nodal points and the average value was recorded. At the end of potential measurements the slabs were broken open and weight loss rods were retrieved. It was immersed in a pickling solution, cleaned, dried and its final weight is obtained. From the weight loss measurements the corrosion rate was calculated.

## RESULTS AND DISCUSSION

**Strength test results:** The strength test results for fly ash blended concrete along with 3 % addition of triethanolamine, diethanolamine, triethylamine and benzimidazole as corrosion inhibiting admixtures are shown in Table-2 and Figs. 1-4.

TABLE-2  
STRENGTH TESTS RESULTS

Tests	Days	C (N/mm <sup>2</sup> )	Stress values (N/mm <sup>2</sup> )			
			TEA	DEA	TE	BEZ
Compressive strength test	3	11.9	13.4	13.7	13.73	13.83
	7	14.6	18.9	16.8	18.01	17.58
	28	25.9	29.3	25.4	26.23	25.57
Split tensile strength	3	1.58	1.64	1.64	1.64	1.66
	7	1.68	1.73	1.72	1.71	1.70
	28	3.03	3.77	3.26	3.46	3.49
Flexural strength test	3	2.89	3.00	2.99	3.16	3.03
	7	3.13	3.13	3.15	3.24	3.19
	28	4.89	4.99	4.71	5.03	4.82
Bond strength test	3	5.43	5.62	5.50	5.52	5.42
	7	6.93	7.13	6.50	7.01	7.11
	28	9.32	10.6	10.4	10.48	10.49

Table-2 shows the compressive strength of fly ash blended cement concrete with and without inhibitors after 3, 7 and 28 days of curing. From the test results, it is found that the compressive strength of the fly ash blended cement concrete is not affected by the addition of 3 % of inhibitors. This value is pronounced for all the inhibitors. Considering the compressive strength from Fig. 1 addition of triethanolamine shows 12.93 % increase in strength, which is the maximum when compared to the control specimen and addition of triethylamine shows 2 % improvement in strength whereas diethanolamine and benzimidazole show the strength equal to that of the control specimen. From Fig. 2, it is observed that the split tensile strength is increased by the addition of triethanolamine, diethanolamine, triethylamine and benzimidazole and they show 24.42, 7.59, 14.19 and 15.18 % improvement, respectively. Fig. 3 shows the results of flexural strength test. Addition of triethylamine shows 13.73 % increase in strength and addition of triethanolamine shows 12.45 % improvement in strength. However, addition of diethanolamine and benzimidazole shows slightly lesser strength by 3.68 and 1.43 %, respectively. From Fig. 4 it is observed that bond strength of fly ash blended concrete specimens with triethanolamine is the maximum by

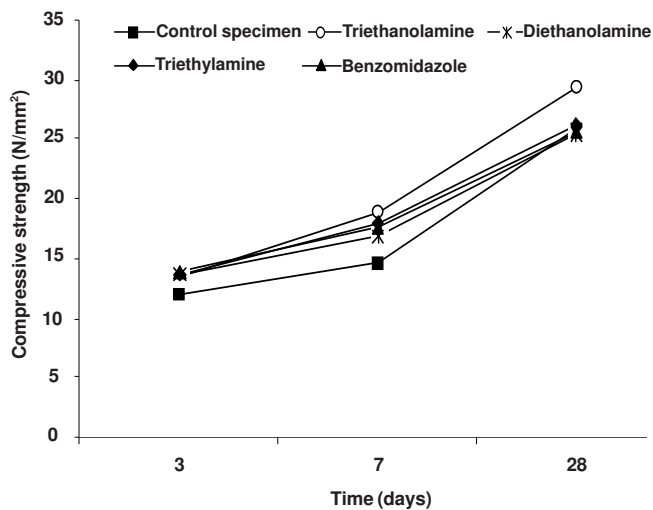


Fig. 1. Compressive strength

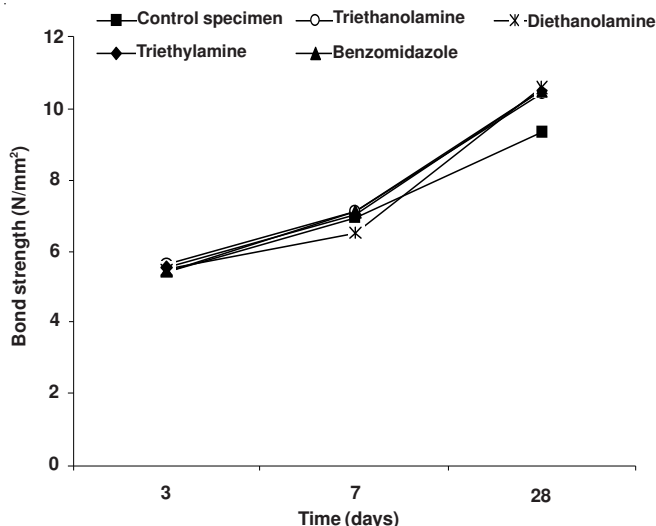


Fig. 4. Bond strength

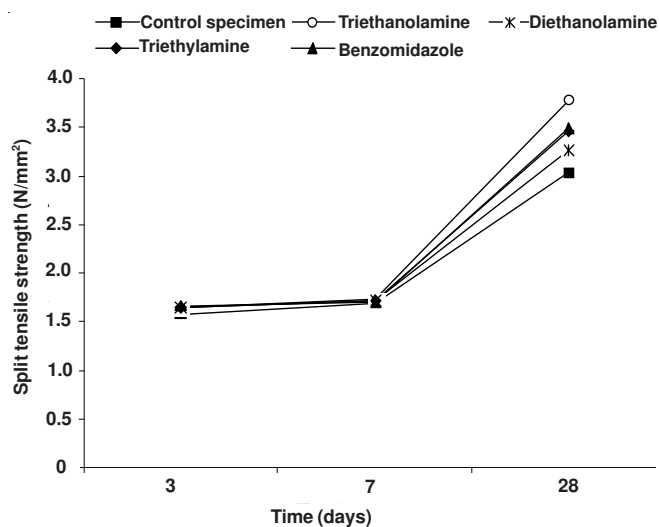


Fig. 2. Split tensile strength

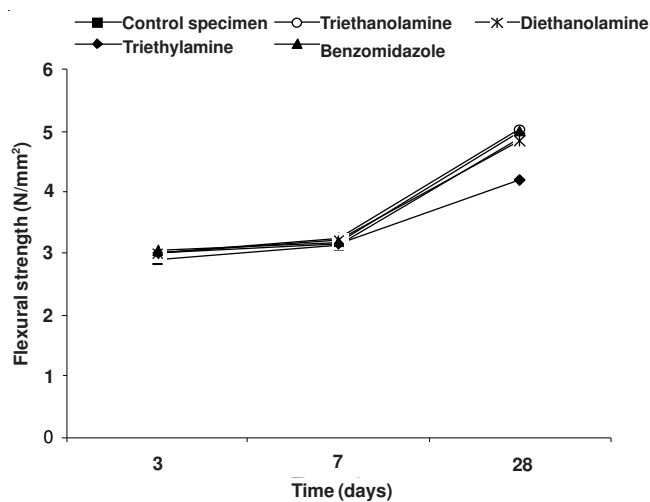


Fig. 3. Flexural strength

13.52 % and diethanolamine, triethylamine and benzimidazole show 11.58, 12.45 and 12.55 % improvement respectively. From the results of the strength tests, it is observed that the strength of fly ash blended concrete has not been affected by the addition of inhibitors and also the inhibitor added specimens display slightly a higher value than the control specimen.

**Micro structural properties:** The micro structural properties such as water absorption, percentage of permeable voids and bulk density values of fly ash blended cement concrete along with various inhibitors are given in Table-3 and Fig. 5. From Table-3, it is observed that, the percentage of water absorption, percentage of permeable voids of fly ash blended cement concrete with all the inhibitors are found to be less when compared to control concrete. The results show that addition of inhibitor has resulted in considerable reduction in water absorption and permeable voids when compared with control specimen. This is because of the transformation of large pores to fine pores as a consequence of the pozzolanic reaction between cement paste and the inhibitors substantially reducing the permeability in the cementitious matrix. By adding inhibitor, bulk density of the specimen is slightly increased due to pore refinement.

Test (%)	C	BEZ	TEA	TE	DEA
Water absorption	4.350	3.547	2.875	3.912	3.899
Permeability	4.721	4.137	3.413	3.814	3.900
Bulk density (g/cm <sup>3</sup> )	2.540	2.575	2.778	2.557	2.600

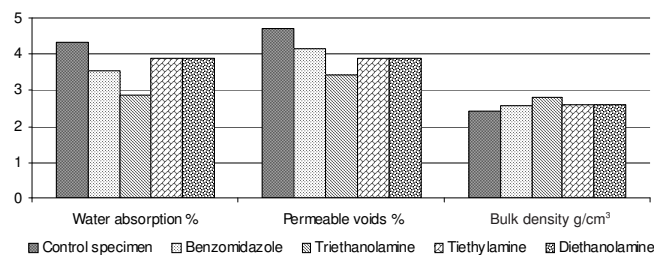


Fig. 5. Micro structural properties

**Diffusion test on concrete disk:** The migration of chloride with application of current density 1 A/m<sup>2</sup> and 3 A/m<sup>2</sup> in presence of four inhibitors are compared. The results are compared with the amount of migration of chloride without application of current. It is observed that under application of current the amount of chloride migrated is more than that without application of current. The result indicates that at 3 A/m<sup>2</sup> the chloride migration is 2.0-2.5 times higher than at 1 A/m<sup>2</sup>.

Time (h)	C	Concentration 100 mM			
		TE	D	TY	B
24	50	267	47	192	1846
48	85	302	267	228	1882
72	120	337	346	228	1633
96	121	444	488	391	1491
120	140	586	572	639	1704
144	170	620	880	796	1710
168	191	657	1065	960	1704
192	192	693	1101	1115	1846
216	227	921	1271	1080	1869
240	298	999	1314	1200	2001
264	298	1083	1385	1364	2165
288	298	1261	1427	1570	2307

All readings are in ppm

C (mmpy)	Corrosion rate (mmpy)			
	TEA	DEA	TE	BEZ
0.170	0.055	0.0455	0.0433	0.0519

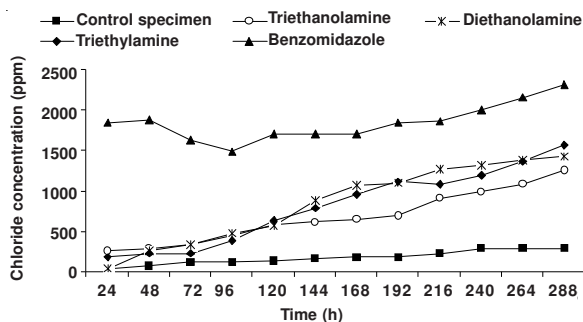


Fig. 6. Chloride diffusion for 100 mM

Time (hours)	C	Concentration 200 mM			
		TE	D	TY	B
24	50	213	213	71	284
48	85	390	497	142	600
72	120	674	852	249	925
96	121	887	1172	426	1005
120	140	1171	1385	533	1100
144	170	1221	1432	650	1810
168	191	1491	1598	852	2100
192	192	1810	1669	1349	2130
216	227	1953	1846	1456	2340
240	298	2094	2023	1526	2550
264	298	2237	2236	1633	2610
288	298	2378	2272	1881	2750

All readings are in ppm

C (mmpy)	Corrosion rate (mmpy)			
	TEA	DEA	TE	BEZ
0.186	0.0709	0.0527	0.0619	0.0500

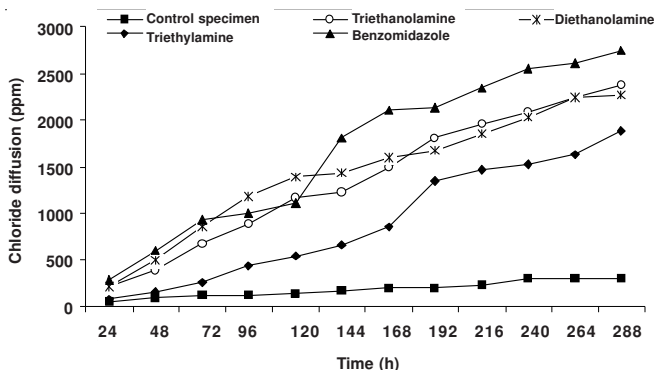


Fig. 7. Chloride diffusion for 200 mM

In Table-8, the potential of rebar immersed different inhibitors at 100 mM concentration is compared with the control system which is without application of current.

Time (h)	Potential measurements for different systems (mV)				
	Control	TEA	DEA	TE	BEZ
72	-569	-568	-586	-568	-573
96	-560	-552	-552	-551	-558
120	-549	-521	-521	-540	-539
144	-543	-485	-485	-512	-510
168	-540	-459	-459	-476	-471
192	-537	-402	-402	-431	-422
216	-535	-358	-358	-396	-335
240	-517	-260	-260	-302	-268
264	-511	-182	-182	-266	-197
288	-503	-104	-104	-209	-126

From the Table-8, it can be seen that there is no much difference in the potential values for the control specimens. For all the other specimens with inhibitors, the potential values of rebar embedded were initially more and finally got reduced. This is due to the fact that the inhibitor ions migrated everyday and the potential of the rebar shifted to more passive direction. Corrosion rate from weight-loss measurement are compared with the potential measurement after 12 days. The corrosion rate reported is average value of 4 rebars embedded in four locations. It is 0.0948 mmpy in the control slab and 0.0426 mmpy in the slab treated with triethanolamine. The corrosion rate is 2.2 times less than in the control specimen. The reduced corrosion rate is mainly because of migration of the triethanolamine inhibitor towards the rebar forming a passive potential layer around it and simultaneously due to the migration of chlorides from inside to outside. This test is a destructive test and hence cannot be used in actual field condition. It is carried out only in the laboratory to establish the efficiency of the inhibitors.

Also the impressed voltage test results show that due to the addition of inhibitors permeability of the concrete was

considerably reduced and the time taken for initiation of corrosion has been increased significantly. It is observed that addition of triethanolamine, diethanolamine, triethylamine and benzimidazole decrease the corrosion rate by 96.76, 73.23, 74.53 and 69.47 %, respectively.

### Conclusion

- The addition of inhibitors as admixture does not show any adverse effects on the strength properties of fly ash blended cement concrete. The specimens with addition of triethanolamine, diethylamine, triethylamine and benzimidazole show slight improvement in the compressive, split tensile, flexural and bond strength when compared with the control specimen without addition of any inhibitor.

- The percentage of water absorption and permeable voids are less when compared with the control specimen.

- The organic inhibitors reduce the ingress of chlorides by filling concrete pores and blocking the porosity of concrete by the formation of complex compounds and reduce the extent of corroded area. From the impressed current test, it is proved that potential values have got reduced by the addition of inhibitors.

- Use of inhibitors in fly ash blended concrete decreases the rate of chloride diffusion. Therefore, the reinforcing steel will be situated in chloride free, highly alkaline concrete. This results a strong re-passivation of the embedded reinforcing steel and thereby the corrosion of the reinforcing steel is halted.

- Among all the inhibitors added, the following is the trend observed on the basis of strength and corrosion resistance properties of fly ash blended cement concrete. triethanolamine > tiethylamine > diethanolamine > benzimidazole.

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