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Chloride Diffusivity and Corrosion Resistance of OPC-MK-RM based Ternary Blended Concrete – An Experimental Investigation

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Abstract. This paper reports the results of the investigation on chloride migration diffusion coefficient and corrosion resistance of metakaolin (MK) and redmud (RM) blended cement concrete with various replacement levels. The concrete had 0% to 14% of metakaolin and redmud as cement replacement separately and with different proportions of metakaolin:redmud (50:50, 60:40, 70:30 and 80:20). The concrete had prepared with water/binder ratios of 0.40, 0.38 0.36 and 0.33. The rapid chloride penetration test (RCPT) was used to calculate the chloride ion (Cl^-) diffusion coefficient (DC) of concrete using the Berke's equation for steady state condition. Corrosion of embedded steel bars in concrete was measured by conducting accelerated corrosion test. The results shows that replacement of metakaolin and redmud was increased the charged passed and diffusion coefficient were digressed. Similar trend was observed in all w/b ratios of concrete blend with different metakaolin:redmud (MK:RM). The percentage loss of weight of embedded steel was lower as percentage of replacement of metakaolin and redmud increased. The concrete of lower w/b ratio has higher value of percentage of loss in weight when compared to concrete which has higher w/b ratio in all replacement levels. In all w/b ratios with percentage replacement of combination of MK:RM, the percentage loss in weight was lower in the proportion of 50:50 compared to 80:20. The results show that the careful choice of composition can reduce the corrosion rate of reinforced steel in concrete.

1. Introduction

India has landmass of 3.28 million km^2. Out of this, 2.42 million km^2 is naturally hard rock. Out of the total hard rock area, approximately 0.7 million km^2 has been identified to contain geological associations and surface shows indicative of possible mineral occurrences. The metallurgical and mineral industries in India generate large quantities of waste materials and by-products every year [20]. By-product disposal and utilization is vividly discussed by solid waste management. The environmental issues related to the by-product wastes and disposal is a noteworthy issue. There is an overall awareness on the changes and utilization of these by-products into construction materials. The use of waste materials helps to improve the microstructure, mechanical and durability properties of mortar and concrete [19].

Unlike by-product pozzolan Metakaolin (MK) has variable composition; in the production process of MK is closely controlled conditions to refine its colour, remove inert impurities and thus higher purity and reactivity can be obtained [4,8]. MK is basically made up of silica and alumina in an amorphous state, these react with calcium hydroxide (CH) produced by Portland cement hydration to form calcium hydrosilicate (C-S-H) and calcium hydroaluminosilicate (essentially gehlenite - C_2ASH_8) [8].

Redmud (RM) is a by-product of the Bayer process, which is used in the production of alumina from bauxite. In each part of alumina produced by Bayer chemical process, about one part of RM is generally discarded as a waste. India generates over 4 million tones of this by-product annually which is not otherwise put to any use. The direction of the researchers undertaken to utilize RM is based on their chemical, mineralogical and physical properties [7]. This superfine particle characteristic of RM is a promising admixture for mortar and concrete [16]. The redmud is fairly caustic, with pH of pastes being typically in the range 11-13. The components of RM are usually considered to be relatively inert and unreactive [9]. As the apparent inertness, lack of reactive silica but its high pH value, the idea of utilizing the pozzolanic reaction to bind redmud mixtures seemed to be a feasible and potentially low cost alternative.

The phenomenon of reinforcement corrosion and consequent damage to concrete is being increasingly recognized as one of the menacing durability problems in reinforced concrete structures. Since the problem of time was identified, considerable research works have been carried out world-wide and a reasonable understanding is now available on the process, mechanism and influencing factors of reinforcement corrosion. Building Research Station, UK said that accelerated normal corrosion is due to the presence of free calcium chloride and stress corrosion takes place in presence of free calcium chloride.

The effect of redmud as corrosion inhibitor of carbon steel embedded in concrete was studied. The result showed that, redmuds are able to maintain steel passivity in the presence of high chloride concentration ie., in mortar 3% $CaCl_2$ by weight of cement [5]. Redmud also effectively acts as corrosion inhibitor of reinforcing steel and constitute a promising way to improve the rebar protection in chloride contaminated mortar and concrete.

This paper reports on a study to evaluate Chloride Migration Diffusion Coefficient and the accelerated corrosion test results of concrete with MK and RM separately and with combinations to the cement replacement. The aim of the work is to determine the diffusion coefficient and the accelerated corrosion test results of concrete in terms of loss of weight of reinforcement in percentage of different water/binder (w/b) ratios.

2. Experimental procedures

2.1. Materials and mix proportions

The following materials were used: Ordinary Portland Cement (OPC) 53 grade (specific gravity: 3.15, Blaine fineness: 320 m^2/Kg), Metakaolin (MK) (specific gravity: 2.54, Blaine fineness: 15 m^2 / gm), Redmud (RM) (specific gravity: 2.74, Blaine fineness: 10.5 m^2 / gm), Fine Aggregate (FA) (specific gravity: 2.60, fineness modulus: 2.964), Coarse Aggregate (CA) (specific gravity: 2.82, fineness modulus: 6.73) and Sulfonated naphthalene formaldehyde condensate based superplasticizer conplast SP 430 (specific gravity: 1.220). The chemical composition of Ordinary Portland Cement (OPC), Metakaolin (MK) and Redmud (RM) are presented in the Table 1.

A concrete mix proportion of water/binder ratios 0.40, 0.38, 0.36 and 0.33 with characteristic target mean compressive strength of 30 MPa, 40 MPa, 50 MPa and 60 MPa are designed without any mineral admixtures. However, the use of several trial mixes is important in the design of concrete. Therefore, to get the optimum proportions, trial mixes were arrived by replacing 0, 2, 4, 6, 8, 10, 12 and 14 percent of the mass of cement by MK and RM in the proportions of 50:50, 60:40, 70:30 and 80:20 respectively. In all the above combinations, a superplasticizer (SP) was used at 1% by weight of the binder for obtaining workable concrete. Mixing is done in the room temperature of $25 \pm 30^{\circ}C$. The composition of the three binder components and mixture details for the concrete with w/b of 0.40 is given in Table 2. For w/b ratio 0.38, 0.36 and 0.33 are referred from the author publication [17,18].

Table 1. Chemical Composition of OPC, MK and RM.

Type	SiO2	Al2O3	Fe2O3	CaO	SO3	MgO	Na2O	K2O	Ig. loss
OPC	21.8	4.8	3.8	63.3	2.2	0.9	0.21	0.46	2.0
MK	52.3	44.9	0.4	0.5	< 0.1	0.2	0.12	0.02	0.8
RM	9.91	20.8	49.0	1.92	0.21	< 0.01	4.11	< 0.01	11.50

2.2. Specimen preparation and curing
The sampling was carried out in accordance with BIS: 1199 - 1959. Each mould was kept in room temperature to preserve the initial moisture condition of the sample; after 24 hours the samples were demoulded and cured in water at $20 \pm 1^{\circ}C$, until testing. The chloride ions passed in the concrete was determined from the average of three test results of 100mm diameter x 50 mm thick concrete specimen. These three 100mm diameter x 50 mm thick concrete specimens were taken from 100mm diameter x 200 mm height concrete cylinder after 28 days of curing by sawing the concrete using a diamond cutter. From the 200 mm height cylinder the top 15mm and bottom 35mm was removed and the remaining portion of cylinder was sawed to three specimens of 50mm thick. The accelerated corrosion test was carried out on 150mm X 300mm cylinders with steel reinforcement (16 mm diameter, 350 mm length have clear cover of 25 mm) was prepared in steel moulds.

2.3. Diffusion coefficient
Diffusion coefficient, Dc of chloride ions increases with the rise in water binder ratio and decreases with the rise of quantity of mineral admixture or additive. Diffusion coefficient tended to increase with increased voltage of 30V in steady state test methods. The effective diffusion coefficient can be calculated from the upstream flux or from the downstream flux of chloride ions, but the result during the steady state was same.
From the RCPT, the charges passed, Q was applied in Berke's Equation (1) to calculate the Chloride Migration Diffusion Coefficient.

$$D_C = 0.0103\,X\,10^{-12}\,X\,Q^{\wedge 0.84}, m^2/s \tag{1}$$

Where
    Dc -   Diffusion coefficient
    Q -   Total charge passed through the concrete specimen

2.4. Corrosion resistance
The experimental test setup arrangement to accelerate the corrosion is given in Figure 1. To create the aggressive environment 3% of NaCl was added in water which acts as electrolyte. Half of the cylinder specimen was immersed in salt water. Regular D.C power supply of 0.2 mA/cm2 is supplied continuously throughout the corrosion period of 15 days. Positive (+ve) terminal of voltmeter is connected with the reinforcement and Negative (–ve) terminal is connected with the copper plate (cathode). Approximate Rate of Corrosion is measured using Digital Multimeter; Specimen is monitored till the completion of Corrosion process. Throughout the corrosion period the specimens were monitored by the half-cell potential survey. The probability of corrosion was gradually increasing day by day.

Table 2. Mix Proportions, w/b = 0.40.

| Mix	Replace- ment %	% Ratio	Cement Kg/m³	MK Kg/m³	RM Kg/m³	FA Kg/m³	CA Kg/m³	SP l/m³	Water l/m³
CC	0 %	--	362.50	--	--	911.53	1044.91	7.44	144.40
MK2	2%	--	355.25	7.25	--	910.16	1044.91	7.44	144.40
RM2			355.25	--	7.25	910.16	1044.91	7.44	144.40
MK4	4%	--	348.00	14.50	--	908.78	1044.91	7.44	144.40
RM4			348.00	--	14.50	908.78	1044.91	7.44	144.40
MK6	6%	--	340.75	21.75	--	907.40	1044.91	7.44	144.40
RM6		--	340.75	--	21.75	907.40	1044.91	7.44	144.40
MR6A		50:50	340.75	10.88	10.88	907.12	1044.91	7.44	144.40
MR6B		60:40	340.75	13.06	8.70	907.07	1044.91	7.44	144.40
MR6C		70:30	340.75	15.23	6.53	907.02	1044.91	7.44	144.40
MR6D		80:20	340.75	17.41	4.35	906.96	1044.91	7.44	144.40
MK8	8%	--	333.50	29.00	--	906.02	1044.91	7.44	144.40
RM8		--	333.50	--	29.00	906.02	1044.91	7.44	144.40
MR8A		50:50	333.50	14.50	14.50	905.66	1044.91	7.44	144.40
MR8B		60:40	333.50	17.40	11.60	905.59	1044.91	7.44	144.40
MR8C		70:30	333.50	20.30	8.70	905.52	1044.91	7.44	144.40
MR8D		80:20	333.50	23.20	5.80	905.45	1044.91	7.44	144.40
MK10	10%	--	326.25	36.25	--	904.64	1044.91	7.44	144.40
RM10		--	326.25	--	36.25	904.64	1044.91	7.44	144.40
MR10A		50:50	326.25	18.13	18.73	903.58	1044.91	7.44	144.40
MR10B		60:40	326.25	21.76	14.50	904.10	1044.91	7.44	144.40
MR10C		70:30	326.25	25.38	10.88	904.01	1044.91	7.44	144.40
MR10D		80:20	326.25	29.01	7.25	903.92	1044.91	7.44	144.40
MK12	12%	--	319.00	43.50	--	903.26	1044.91	7.44	144.40
RM12		--	319.00	--	43.50	903.26	1044.91	7.44	144.40
MR12A		50:50	319.00	21.75	21.75	902.73	1044.91	7.44	144.40
MR12B		60:40	319.00	26.10	17.40	902.62	1044.91	7.44	144.40
MR12C		70:30	319.00	30.45	13.05	902.52	1044.91	7.44	144.40
MR12D		80:20	319.00	34.80	8.70	902.41	1044.91	7.44	144.40
MK14	14%	--	311.75	50.75	--	901.88	1044.91	7.44	144.40
RM14		--	311.75	--	50.75	901.88	1044.91	7.44	144.40
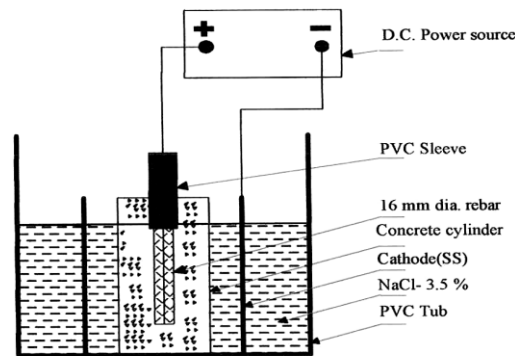
Figure 1. Experimental test setup arrangement.

3. Results and discussions

3.1. Diffusion coefficient

The chloride migration diffusion coefficient (Dc) of concrete blends with MK, RM and MK:RM proportion of w/b ratio 0.40, 0.38, 0.36, and 0.33 was calculated based on RCPT value by using Berke's equation. The calculated chloride diffusion coefficient was plotted against the various mix designation (% replacement). Figures 2 to 4 show the diffusion coefficient values of different w/b ratios of concrete blended with MK, RM and MK:RM. It can be seen that the diffusion coefficient, Dc of concrete with MK for all w/b ratio 0.40, 0.38, 0.36 and 0.33 continuously decreases with increase in replacement level of MK. Also it is noted that, in all replacement levels the diffusion coefficient of w/b ratio 0.40 was higher than other three w/b ratios. Lesser diffusion coefficient was observed when the concrete grade was enhanced. This may be due to the effect of particle packing of the mix. In this present study it was observed that in all w/b ratios the MK concrete has lesser diffusion coefficient and controlled concrete has a higher value.

In all w/b ratios, concrete blend with RM, the diffusion coefficient values decreased up to 10% replacement of cement with RM and thereafter increased. Hence the value is quiet less compared with controlled concrete. For all w/b ratios, whereas concrete blend with MK:RM compared with controlled concrete, the diffusion coefficient decreased with increase in percentage replacement level of MK:RM. The MK:RM blended concrete have denser microstructure than controlled concrete, and it will not allow the chloride ion to penetrate easily because of good impermeability characteristics.

The diffusion coefficient of controlled concrete with w/b ratio 0.33 is 1.54 times less when compared with w/b ratio 0.40. The diffusion coefficient of concrete at 8% replacement with MK alone with w/b ratio 0.33 is 1.53 times less than concrete compared with w/b ratio 0.40. Similarly, the diffusion coefficient of concrete at 4% replacement with RM alone with w/b ratio 0.33 is 1.59 times less than concrete compared with w/b ratio 0.40. Whereas, at 10% replacement of concrete with combination of MK:RM of ratio 50:50 and 80:20 with w/b ratio 0.33 is 1.56 and 1.59 times less than the concrete when compared with w/b ratio 0.40. The results indicate that the replacement with MK and RM influence the concrete durability characteristics. The effective chloride diffusion coefficients of PC, PC-15% PFA and PC-15% MK paste of w/b ratio 0.4. Both the PC-MK and the PC-PFA pastes gave lower chloride diffusion coefficients than the PC paste, and the former gave particularly low values [6].

The chloride diffusion up to 365 and 1095 days was determined by [2, 10]. They reported that the apparent diffusion coefficients were reduced on increasing the exposure time and on decreasing the w/b ratio, and showed marked decreases with increasing metakaolin content.
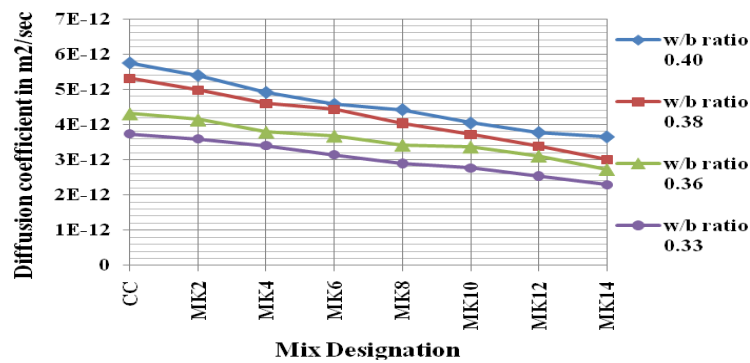
Figure 2. Variation of Diffusion coefficient of concrete for all
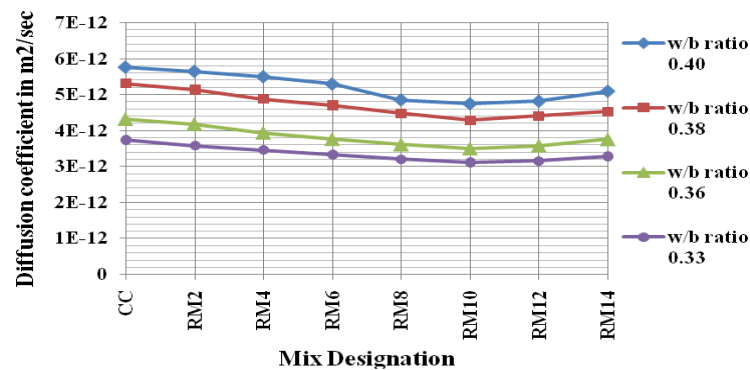w/b ratios with metakaolin.

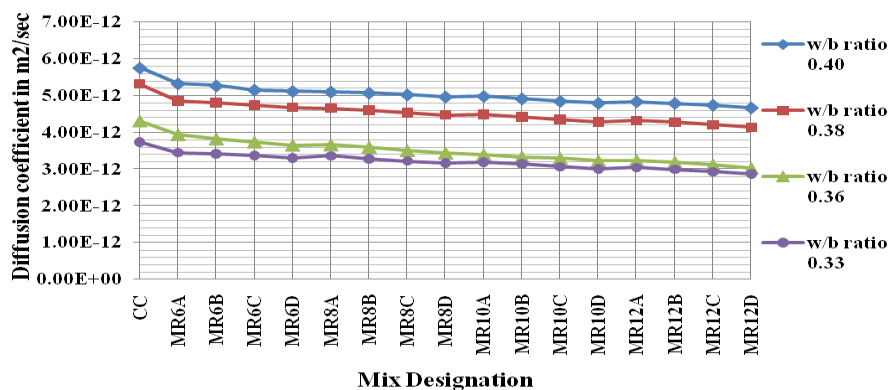**Figure 3.** Variation of Diffusion coefficient of concrete for all
w/b ratios with redmud.

Figure 4. Variation of Diffusion coefficient of concrete for all w/b ratios
with MK-RM blend.

3.2. Accelerated corrosion

In general, there are two main focuses for the study of steel corrosion in concrete: passivation of steel in a certain alkaline condition, and the acceleration effect of chloride ions in pore solution. Due to the high alkalinity of concrete pore fluid, steel in concrete initially and, in most cases, for sustained long periods of time, remains in a passive state. Initiation of corrosion is either due to reduction in alkalinity arising from carbonation and attack of chloride ions causes the breakdown of the passive layer. In this investigation, the percentage loss of weight of steel reinforcement was calculated by

accelerated corrosion test. The initial weight of steel rod was taken before the preparation of the specimen. After the corrosion period of 15 days the steel rod was taken from the concrete and weight was measured. Finally the loss in weight in grams and loss of weight in percentage were calculated. The influence of MK, RM and MK-RM blend on accelerated corrosion in terms of loss of weight of reinforcement in grams are shown in Figures 5 to 7.

Figures 5 show the variation of loss of weight of steel reinforcement in grams of concrete of all w/b ratios (0.40, 0.38, 0.36 and 0.33) with metakaolin as partial replacement with cement. It is observed that the corrosion rate and loss of weight are gradually reduced compared to the controlled concrete with the increasing % replacement of metakaolin. The maximum compressive strength was obtained for mixes with 8% replacement of cement by metakaolin in all w/b ratios. In the w/b ratio 0.40, 0.38, 0.36 and 0.33, the percentage loss of weight measured at 8% replacement was 4.13, 4.16, 3.51 and 3.30 respectively whereas, for the controlled concrete of same w/b ratios are 10.33, 9.48, 8.40 and 7.77. This indicated that the replacement of metakaolin refined the pores and thereby the permeability and corrosion get reduced. It is also noted that beyond 10 % replacement level the percentage loss of weight was increasing.

Figures 6 show the variation of loss of weight in grams of steel reinforcement in concrete of all w/b ratios (0.40, 0.38, 0.36 and 0.33) with redmud as partial replacement. It can be seen that the percentage of loss of weight is decreased by increasing the percentage replacement of redmud compared to controlled concrete. The maximum compressive strength is obtained for mixes with 4% replacement of cement by redmud in all w/b ratios. The corrosion rate and loss of weight in % was gradually decreased up to 10 % replacement level of redmud and thereafter it was increased. For the w/b ratio 0.40, 0.38, 0.36 and 0.33, the % loss of weight at 4% replacement of redmud was measured 4.65, 3.05, 3.05 and 3.41; at 10% replacement of redmud was measured 2.03, 1.72, 1.57 and 1.61 respectively. For controlled concrete of same w/b ratios the values are 10.33, 9.48, 8.4 and 7.77 respectively. It is also noted that the % of loss in the weight of steel in concrete with redmud as replacement is very low compared to the concrete with metakaolin in all replacement levels. The result shows that the redmud is able to maintain steel passivity by forming a passive layer on the steel reinforcement. Concrete with redmud as replacement, due to its high pH, leads to high alkalinity which protects steel by forming a passive film that hinders corrosion.

The variations of loss of weight in grams of metakaolin-redmud blends as replacement in concrete of all w/b ratios (0.40, 0.38, 0.36 and 0.33) at 28 days curing with different MK:RM ratios (50:50, 60:40, 70:30 & 80:20) are given in Figures 7. In all the mix proportions and all w/b ratios, it is observed that the percentage of loss of weight of steel reinforcement in concrete decreased with the increase in percentage replacement of MK-RM blends. It is also observed that in MK:RM ratio of all w/b ratios the loss of weight in the percentage of steel reinforcement in concrete of the ratio 50:50 is lower compared to the ratio 80:20. Increase in the amount of redmud in all the ratios shows that it can maintain steel passivity by forming a passive layer on the steel reinforcement. In w/b ratio 0.40, the loss of weight in percentage of 10% replacement of MK-RM blend at 50:50 was 4.91, whereas for the ratio 80:20 was 6.11 and these values are lower than controlled concrete value of 10.33. Similarly in w/b ratio 0.38, loss of weight in 10% replacement of MK-RM blend at 50:50 was 4.07, whereas for the ratio 80:20 was 5.16 and these values are lower than controlled concrete value of 9.48. In w/b ratio 0.36, the loss of weight in percentage of 10% replacement of MK-RM blend at 50:50 was 3.60, whereas for the ratio 80:20 was 4.49 and these values are lower than controlled concrete value of 8.40. In the w/b ratio 0.33, the loss of weight in 10% replacement of MK-RM blend at 50:50 was 2.98, whereas for the ratio 80:20 was 3.88 and these values are lower than controlled concrete value of 7.77. The effect of metakaolin on the corrosion resistance of cement mortar specimen when exposed to the corrosive environment of total immersion in 3.5% NaCl solution was studied by [2]. It was reported that the use of metakaolin as a cement replacement up to 10% improved the corrosion behaviour of mortar specimens, while there is no positive effect when metakaolin was added in greater percentage. The redmuds are good inhibitors of the chloride attack and also effective corrosion inhibitors of reinforcing steel embedded in chloride contaminated mortar conclude by [5]. According to the obtained results, it can be said that redmuds constitute a promising way to improve the rebar protection in chloride-contaminated concrete.
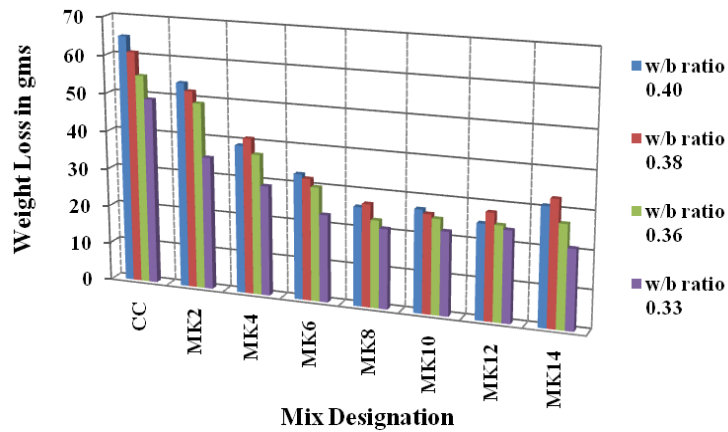
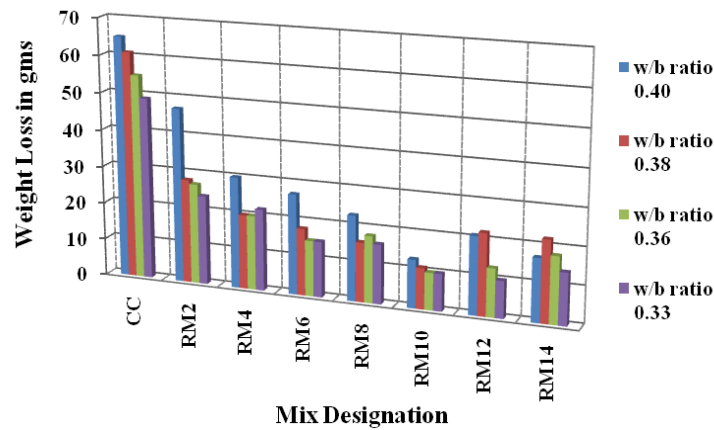**Figure 5.** Influence of MK on corrosion of steel reinforcement in concrete for all w/b ratios.

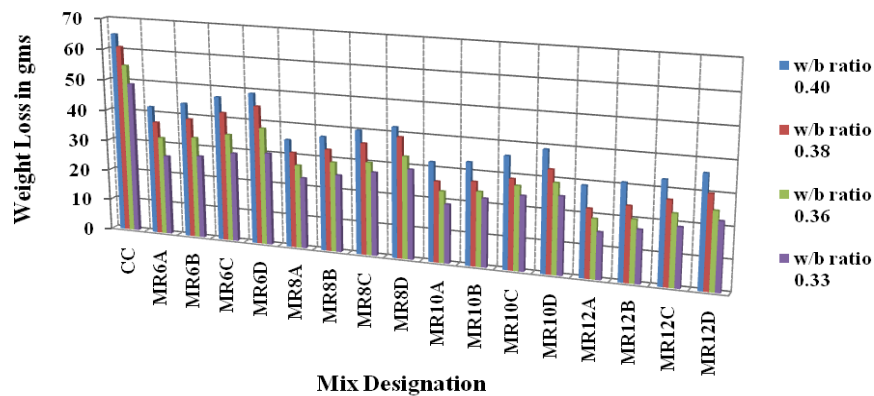**Figure 6.** Influence of RM on corrosion of steel reinforcement in concrete for all w/b ratios.

Figure 7. Influence of MK-RM blend on corrosion of steel reinforcement in concrete for all w/b ratios.

4. Conclusions

On the basis of the investigation of diffusion coefficient and accelerated corrosion test of concretes containing metakaolin, redmud and a combination of metakaolin and redmud, the following conclusions have been drawn.

- − The chloride ion diffusion coefficient, Dc of concrete increases with the rise of w/b ratio. The diffusion coefficient may decrease with the rise in quantity of metakaolin or redmud or metakaolin-redmud blend. All blended concrete have high resistance to diffusion of chloride ions. The metakaolin and redmud may improve the distribution of pore size and pore shape of concrete. With an increase in w/b ratio, more pores and diffusing paths may form, and the resistance to chloride ions decreases, so the chloride ion diffusion coefficient increases from 5.76 E-12 for controlled concrete to 4.79 E-12 for MK:RM blend concrete.
- − There is significant reduction (example: at w/b ratio 0.33, from 7.77% to 3.93%) in loss of weight due to corrosion of reinforcement when metakaolin is added in concrete mixes, beyond 10% replacement. A greater reduction (ex: at w/b ratio 0.33, from 7.77% to 1.56%) in loss of weight of reinforcement was found when redmud is added in concrete mixes. The % of loss in the weight of steel in concrete with redmud as replacement was very low (1.56%) compared to the concrete with metakaolin (3.27%) in all replacement levels. The result shows that the redmud is able to maintain steel passivity by forming a passive layer on the steel reinforcement.
- − A systematic reduction in loss of weight of reinforcement was found when metakaolin - redmud blend replaced cement. It also been noted that the redmud is acting as a good corrosion inhibitor of reinforcing steel in all concrete with higher redmud replacement compared to metakaolin, which shows that redmud constitute a promising way to improve the rebar protection in concrete. From the above conclusions it is recommended to use MK and RM blend for the purpose of durability.

Acknowledgement

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