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Thermodynamics for Polycarboxylate Macromolecule Interaction with Potassium Ferrocyanide and Applications to Portland Cement Paste Compressive Ability

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Abstract: The effect of Sika Viscocrete (Polycarboxylate- based macromolecule polymer) on the equilibrium thermodynamic of $K_4Fe(CN)_6$ in neutral potassium chloride and its application for OPC (Ordinary Portland Cement) paste were added. Portland cement is manufactured from raw limestone, clay, mudstone, and shale deposits. The cyclic voltammetry of Sika Viscocrete with different concentrations with $K_4Fe(CN)_6$ at 290K was measured using a potentiostat, delivered from the USA in KCl (0.1 M) as the medium, glassy carbon electrode, platinum wire electrode, and standard Ag/AgCl electrode are used. Different solvation parameters were evaluated by using various scan rates. Also, the mechanisms and the stabilization of the complexes formed are discussed. The compressive (compressive ability) strength of the cement mixture with water at curing times (1, 3, 7 and 28 days) was determined by using different percentages of polycarboxylate-based macromolecule as an environmental application. The results indicated that Viscocrete is combined with the iron present in the cement and increase the compressive ability using the concentrations of macromolecule material, 0.25% and 0.5% then decreases with the concentrations 0.75% and 1%.

Keywords: Polycarboxylate- based macromolecules, Cement (P. Portand), solvation properties, equilibrium thermodynamics, stability constant, Gibbs energies of interaction, strength of compressibility (compressive ability).

1.Introduction

Cement increases its hardness in aqueous solution and form concrete. The aggregates are hold together to form concrete using aqueous products that act as a binder [1]. Its production cost is quite cheap and very important in many industries.

After hydrographical examinations, limestone, clay, mudstone, and shale deposits are used to make ordinary Portland cement, then heated in a kiln to 1450°C in a calcination process, liberating carbon dioxide forming CaO, then combines chemically with some of the existing materials in the mixture. After that calcium silicates with cement compounds are formed [2]. Clinker is formed as hard substance. Finally, ordinary Portland cement is made of grinding clinker with a small amount of gypsum.

Sika Viscocrete (poly carboxylate-based macromolecule, polymer) is a chemical admixture used as a superplasticizer to produce fast hard material. Also using water to form high strength in the concrete and gives excellent slump retention for prolonged times [3, 4]. Especially increasing its compressibility ability and decreasing iron oxidation. In the present work, we studied the effect of Sika Viscocrete (Superplasticizer), a polycarboxylate- based polymer, on improving the mechanical properties of concrete such as compressive strength at different dosages (0.25%, 0.5%, 0.75% and 1%). There are no chemical reactions between viscocrete SP and used cement. The physical reactions are only take place in mixture with low water ratio [4]. Increasing the admixture dosages to 0.25% and 0.5% which leads to increase in the compressibility ability. On the contrary, increasing the admixture dosages up to 0.5% such as 0.75% and 1% leads to decrease in compressive strength values due to high water reduction by Sika Viscocrete (poly carboxylate based polymer) [5,6]. The effect of Sika Viscocrete macromolecule on solvation of $K_4Fe(CN)_6$, which is prepared from hematite, can be studied [7]. We found from this study, Sika Viscocrete superplasticizer interact with iron ions [8.9].

2. Materials and methods

2.1 .Potassium Ferrocyanide

$K_4[Fe(CN)_6]3H_2O$ [10], BHD was used.

2.2. Ordinary P. Cement OPC is provided from Suez Cement Company, Suez, Egypt; with known chemical composition [11, 12]. The phase composition of the cement was evaluated using Bough's equation.

2.3. Sika Viscocrete (poly carboxylate based polymer)

Sika Viscocrete (poly carboxylate based polymer) has a high molecular mass (M_r = 100 000). It consists of acrylic acid or copolymers of acrylic acid and maleic acid. Sika Viscocrete (a polycarboxylate based polymer) is suitable plasticizer for concrete admixture [13]. The properties of Sika viscocrete are shown in (Table 1) [14] .The CAS details of the chemicals used is given in Table 2.

The important target here in our studies is to explain the different properties of the sample description given in Table 2.

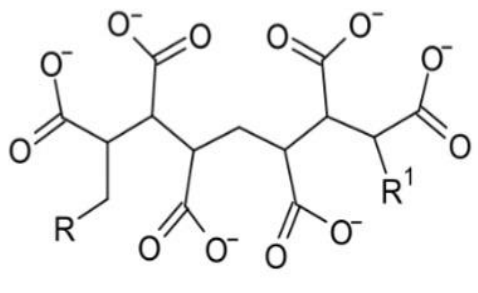

Fig.1: Polycarboxylatepolymer.

Table 1: Properties of Sika Viscocrete (poly carboxylate based polymer)

Property	Description
Form	Liquid
Color	Clear
Relative Density	1.080
pH Value	4.3
Chloride Content %	<0.1 Chloride Free
Alkali Content %	0.6

Table 2: CAS of chemicals used.

Compound	CAS	Chemical Name
Sika.ViscoCrete.3425	2634-33-5	Aqueous solution of polycarboxylate
Ordiary Portlend Cement (OPC)	65997-15-1	Cement Portland Chemical
Potassium ferrocyanide trihydrate	14459-95-1	Potassium hexacyanoferrate
Potassium chloride	7447-40-7	Potassium chloride

2.4 .Cyclic Voltammetry

The voltammogram was developed by using a potentiostat DY2000 apparatus choosing various scan rates. In practical work, deionized water was used. The Ag/AgCl electrode put in saturated KCl is applied as reference electrode [15, 16] .A Pt wire was used as counter inert electrode. The working glassy carbon electrode was prepared in our laboratory, polished with wool piece, and finally cleaned by washed with deionized water [17].

2.5. Water reduction percentage (%)

Standard water of consistency values of all cement mixtures with water were measured according to ASTM methods using Vicat apparatus [18, 19]. The cement paste was filled into the Vicat mold. Then the paste in the mold was vibrated to make the surface of the paste free from air bubbles. To measure the amount of water needed to produce a paste of standard consistency, the Vicat plunger must be settled to a point of 5 to 7 mm from the bottom of the Vicat mold [20].

2.6. Preparing the cement paste and Molding

The standard water of consistency is used for preparing fresh pastes, cast in one-inch cubic molds. We remove air bubbles and compact the molded samples [21].The moulds were cured for 24 hours [22].

2.7. Compressive (Compressibility) Ability and Phase Composition

Compressive (Compressibility) ability values for 3 hardened cubes of each dose were determined by using Ton industry machine according to standard ASTM specifications. These measurements occurred at each curing times. The measured values refer to increase in the mechanical properties at each curing time [23, 24].

3. Results and Discussion

The redox processes of Fe (II) ions in the investigated samples was experimentally studied [25-28]. The cyclic voltammogram obtained are analyzed by the use of equations mentioned in the previous research paper [29].

3.1. Cyclic voltammetry of $K_4Fe(CN)_6$:

The CV of 14.3 mM$K_4Fe(CN)_6$ was measured at 290 K (Fig.2). Redox reaction mechanism has been explained in detail in the previous research [29].The standard transfer rate constant [30-32], the molecular thermodynamic Gibbs free energies of complexation [33- 37], the molecular thermodynamic parameters for the interaction of Viscocrete (poly carboxylate based polymer) with $K_4Fe(CN)_6$ in 0.1 M (molar) potassium chloride solution are calculated [29].

3.2. CV (Cyclic Voltammetry) of $K_4Fe(CN)_6$ in presence of Sika Viscocrete in 0.1M potassium chloride following (First waves).

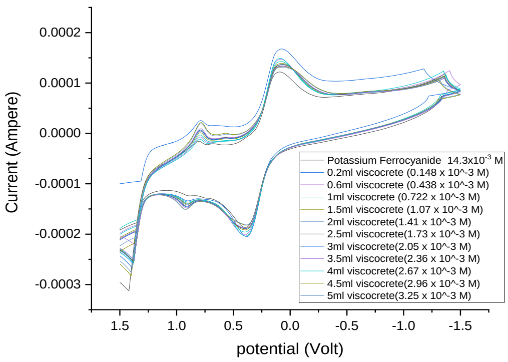

Fig. 2. Effect of the addition different concentrations of sika viscocrete on potassium Ferrocyanide (14.3 x 10^{-3}M or 14.434x10^{-3}m, molal)[1] on the first couple wave of C. at 290 K and scan rate 0.1V.s^{-1}

The effect of the addition of Sika Viscocrete polymer with different concentrations to 14.3(mm) mille mole potassium ferrocyanide on CV results were studied.The analysis data are presented and illustrated in previous research [29]. We noticed increase in electrode potential differences, anodic diffusion coefficient, cathodic diffusion coefficient, electron transfer rate constant, cathodic surface coverage, amount of electricity, anodic surface coverage and the part of anodic quantity of electricity proving the complex reaction occurred by the interaction of polymer with ferrocyanide ions. These data were obtained by analysis of the first peak couple.

The data show standard uncertainties, for μ (E_{pa},E_{pc}) = 0.0148V**,** μ (k_s, electron transfer rate constant)= 5.882x10^{-6} cm.Sec^{-1},diffusion coefficients (Da ,Dc) = 1.476x10^{-12} $cm^2.S^{-1}$**,** μ (surface coverage), Γ_c& Γ_a=2.093x10^{-8} mol.cm^{-2}, μ (quantity of electricity, Qc &Qa =1.415 Coulomb C).

Gibbs free energies and thermodynamic parameters stability constants of interaction of potassium ferrocyanide with Sika viscocrete polymer were estimated and found to be in the range of electrostatic interaction that means physical interaction following the first peak couple (Table 3). The same Analysis for the effect of the polymer used on potassium ferrocyanide was done for the second couple of peaks as explained in Fig.3.

Table 3: Stability constants for potassium Ferro cyanide with sika viscocrete complex and the interaction free energies (ΔG) following 1^{st} wave couples.

[M] x10^2 mol.L^{-1}	[P] x 10^3 mol.L^{-1}	($E_{p,1/2}$)M	(Ep,1/2)P	ΔE mV	J (L/J)	log βj	ΔG (KJ/mol)
1.42	0.148	0.318	0.333	-0.015	0.01	-0.22	1.226
1.40	0.438	0.318	0.333	-0.015	0.03	-0.15	0.864
1.39	0.722	0.318	0.335	-0.017	0.05	-0.13	0.73
1.37	1.07	0.318	0.331	-0.013	0.07	0.006	-0.035
1.35	1.41	0.318	0.335	-0.017	0.10	0.002	-0.01
1.33	1.73	0.318	0.336	-0.018	0.13	0.047	-0.26
1.32	2.05	0.318	0.323	-0.005	0.15	0.33	-1.85
1.30	2.36	0.318	0.327	-0.009	0.7	1.68	-9.36
1.28	2.67	0.318	0.345	-0.027	0.8	1.59	-8.85
1.27	2.96	0.318	0.328	-0.01	0.9	2.10	-11.6
1.25	3.25	0.318	0.322	-0.004	1	2.42	-13.46

Standard uncertainty for, μ for interaction energies between the polymer and potassium ferrocynide following for the first wave couple = 0.093 K.J.mol^{-1}.

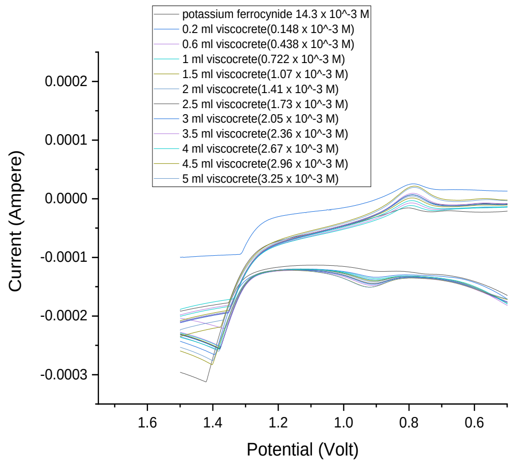

Fig. 3 Effect of addition different concentrations of Sika Viscocrete on potassium Ferro cyanide (14.3 x $10^{\wedge -3}$M or 14.434 x10^{-3} mole /kg) on CV at 290 K and scan rate 0.1V.S^{-1}. (Second couple of waves)

The effect of Viscocrete polymer concentration on the CV of the ferrous ions in potassium ferrocyanide is also studied and thermodynamic parameters calculated using the second couple of peaks represented in Figure 3

The data show standard uncertainties, for μ (E_{pa}, E_{pc}) =0.014V, μ (k_s, electron transfer rate constant) = 6.1x10^{-6} cm.Sec^{-1}, diffusion coefficients (Da, Dc) = 1.5x10^{-12} cm^2.S^{-1}, μ (surface coverage), Γ_c& Γ_a=2.0x10^{-8} mol.cm^{-2}, μ (quantity of electricity, Qc& Qa)=1.2 Coulomb (C).

Table 4: Stability constant for Potassium Ferrocyanide with Sika Viscocrete complex and the interaction free energiesfor the second wave couple.

[M] x10^2 mol.L^{-1}	[P] x 10^3 mol.L^{-1}	(Ep,1/2)M	(Ep,1/2)P	ΔEmV	J (L/J)	log βj	ΔG (KJ/mol)
1.42	0.148	0.861	0.873	-0.012	0.0104	-0.1682	0.9361
1.40	0.438	0.861	0.872	-0.011	0.0312	-0.0859	0.4782
1.39	0.722	0.861	0.873	-0.012	0.052	-0.0447	0.2488
1.37	1.07	0.861	0.874	-0.013	0.078	0.0064	-0.0354
1.35	1.41	0.861	0.882	-0.021	0.104	-0.0675	0.3754
1.33	1.73	0.861	0.883	-0.022	0.13	-0.0225	0.1251
1.32	2.05	0.861	0.890	-0.029	0.156	-0.0835	0.4647
1.30	2.36	0.861	0.882	-0.021	0.7	1.4744	-8.2052
1.28	2.67	0.861	0.868	-0.007	0.8	1.9379	-10.7844
1.27	2.96	0.861	0.896	-0.035	0.9	1.6688	-9.2868
1.25	3.25	0.861	0.898	-0.037	1	1.8466	-10.2767

3.3. Water reduction %:

Reduction in H_2O is considered one of the main parameters that show the efficiency of the admixtures. (Table 5) shows the values of water/cement (W/C) percentages and water reduction percentage, of admixed cement pastes with Sika Viscocrete-3425 super plasticizer. It was observed that all admixed cement pastes have a lower water to cement ratio W/C than the blank sample due to the adsorption of the Viscocrete used on the cement. It induces a steric hindrance due to a long side chain in the poly carboxylates-based super plasticizer and preventing particle contact [38].The results also show the increase in the dosage of Viscocrete is followed by an increase in the water reduction percentage. The water reduction values .reached 26.47, 29.41, 30.39 and 33.33% when Visco Crete was added by 0.25, 0.50, 0.75 and 1.00 wt. % respectively, which reflected the effect of this material.

3.4. Compressive (compressibility) ability of the various cement mixture containing various amounts of Viscocrete.

The compressive (compressibility) ability of the hardened cement mixture with different percentages from Viscocrete are given in (Table 6) and in (Fig. 4).

Water of Consistency and Water reduction %:

Table 5: Water/cement ratios and water reduction% of the neat and admixed cement pastes containing different doses of the used admixture (Viscocrete).

Paste	Additive dosage (%)	Water of consistency (ml)	W/C ratio	Water reduction (%)
Neat	0	102	0.255	0
Viscocrete	0.25	75	0.1875	26.47
	0.50	72	0.180	29.41
	0.75	71	0.1775	30.39
	1.00	68	0.17	33.33

Table 6: Compressive strength values of the various cement pastes containing different doses of Viscocrete after 1, 3, 7, 28 days of hydration.

Admixture doses	Compressive strength (MPa)			
	1 day	3 days	7 days	28 days
0%	38.4	70.4	76	92.4
0.25%	45.07	74.45	83.68	94.64
0.5%	71.63	78.88	91.36	108.48
0.75%	54.29	64.32	65.76	85.12
1%	59.79	70.29	73.36	88.88

The reduction in standard water of consistency is considered one of the main parameters that show the efficiency of the admixtures. (Table 5) shows ratios of W/C different percentages of Sika Viscocrete. It was observed that all admixed cement pastes have a lower W/C ratio than the blank (neat) sample. This is due to Viscocrete effective dispersal properties. It is adsorbed on the cement. It induces a steric hindrance due to a long side chain in the poly carboxylates-based super plasticizer, preventing particle contact [38]. Also, an increase in the dosage of Viscocrete is followed by an increase in the water reduction percentage. The water reduction values reached 26.47, 29.41, 30.39 and 33.33% when Visco Crete was added by 0.25, 0.50, 0.75 and 1.00 wt. % respectively, which reflected the effect of this super plasticizer.

3.5. Compressive (Compressibility) ability values of the cement mixture with water for various percentages of Viscocrete.

In (Table 6), the given compressive (compressibility) affinity values of hardened cement mixture with macromolecule with different percentages are shown and then graphically represented in (Fig. 4).

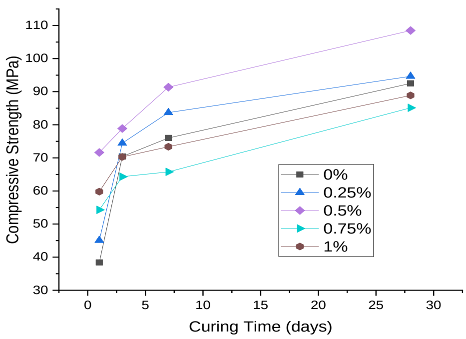

Fig.4: The relation between Compressive strength of of various cement pastes containing different doses ofViscocrete with curing time (1, 3 ,7 , 28 days)of hydration.

The mechanical properties such as compressive (compressibility) ability values of hardened cement paste containing various percentages of Sika Visco Crete are shown in (Table 6) and represented in (Fig. 4). In general, increasing curing time up to 28 days is associated with continuous improvement in compressibility ability values for the applied samples. This was referred to a progressive hydration process of cement grains which forms different binding phases such as C-A-S-H) and (C-A-H) [39]. Interestingly, after 1-day, it was observed that adding different percentages of 0.25, 0.50, 0.75 and 1.00 wt. % Visco Crete (poly carboxylate based polymer) increases the values of compressibility ability, strength of hardened. This shows the acceleration effect of Viscocrete on the hydration process. Also, adding ViscoCrete-3425 leads a decreasing in total porosity. Thus, porescan be filled with the smallest number of binding phases, forming a compact structure. According to the data, adding 0.25 and 0.50 weight percent increases compressive strength by 2.42 and 17.4%, respectively. When the dosage is increased to 0.75 wt% and 1.00 wt. %, the values of compressive strength decrease respectively by 7.88 and 3.81%, corresponding to the sample. This may be due to increasing the dosage of Visco Crete causing decrease water content. In addition, increasing the dose reduces the cohesiveness. The interaction for polycarboxylate polymer with the Na_2O oxide, as an example, which can be done for other oxides in is illustrated as given in Fig.4.The binding force or strength of the carboxylic acid group (-COOH) with different metal ions like ferrous ones are well known. Therefore using polycarboxylate polymer increase the interaction between different oxides in cement paste and also the combination with iron found in rods in the concrete.

Conflict of interest:

There is no conflict of interest with any body about this work.

Conclusions:

The $K_4Fe(CN)_6$ reaction show four peaks. Two couples of peaks are appeared. By adding Sika Viscocrete macromolecule more developing of the second peaks were observed. In potassium $K_4Fe(CN)_6$ analysis, most Ipa/

Ipc data are smaller than one .By more increasing in $K_4Fe(CN)_6$,the anodic and the other surface coverage $\Gamma_{c,}$ $\Gamma_a$are increased indicating small association. Increasing in Sika Viscocrete Polymer added to potassium ferrocyanide composition lead to increase in the Gibbs free energies and stability constants of the interaction between our compounds indicating more complexation.

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