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DETERMINATION OF CRITICAL VALUE OF pH OF GEL FORMATION IN SILICATE-ALKALI MIXTURES OF VARIOUS COMPOSITION

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Abstract

Purpose: *The aim of this study is the research of a possibility of a direct and reverse titration usage for the critical pH determination. pH value change of the silicate-alkali mixture was analyzed while addition of alkalis, salt water and sodium hydrogen carbonate solutions.* ***Methods:*** *Methods of direct and reverse titration were used for the determination of various additives influence on results of pH measuring of sodium silicate mixtures.* ***Results:*** *Additives method or reverse titration of the alkali solution are the most appropriate methods for the determination of free alkali content in silicate solutions. Dilution of silicate-alkali solutions by salt water or its model leads to the faster decrease of pH value, than in case of water without any salts of hardness. Hydrogen carbonates influence should be accounted when execution of accurate calculations used in the development of silicate-alkali mixtures usage technology.* ***Discussion:*** *Alkali-silicate mixtures would be solutions for a long time when filtration proceeds in the porous medium at* $pH > pH_{crit}$*. The time of such solutions existence must be characterized by a before inductive period. When reaching the critical pH value, a gel is formed during an inductive period or the time of gel formation. As a rule, the before inductive period is in ten times or even more greater than the time of gel formation.*

Keywords: dynamics of pH change; gel formation; pH value; sodium silicate.

1. Introduction

Mixtures based on sodium silicate are true solutions in basic environment with viscosity close to the viscosity of water. Solution can turn into gels at certain conditions. In this case the magnitude of viscosity will increase in several orders. The possibility of such transition is limited by two factors. They are critical concentration of gel formation $C_{crit.}$ and critical value of pH of gel formation $pH_{crit.}$

Critical concentration of gel formation is the maximum concentration of sodium silicate, when rate of gel formation is equal to zero. Similarly pH_{crit} is the minimal value of pH, at which gel formation does not proceed in basic medium [1].

2. Analysis of the latest research and publications

It is possible to adjust the process of gel formation of silicate mixtures in porous medium because of presence of critical value of pH and ability of layer of ore to neutralize the alkali

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while alkali solutions filtration. Regulation is executed by changing the concentration of sodium silicate, mineralization and usage of various additives, which slow down the process of gel formation [2]. The most convenient way of gel formation process regulation is regulation the value of pH of silicate mixtures by addition of alkali or acid solutions into these mixtures [3].

Characterization of the alkali addition influence on the change of pH value.

Sodium silicates are sodium salts of very weak diprotic silicic acid H_2SiO_3. That is why silicate solutions have bright alkali reaction.

On the basis of silicic acid formula its two stage ionization will proceed according to a scheme [4, 5]:

$$H_2SiO_3 \overset{K_1}{=} HSiO_3^- + H^+; \quad \log K_1 = -9{,}80$$

$$HSiO_3^- \overset{K_2}{=} SiO_3^{2-} + H^+; \quad \log K_2 = -12{,}16$$

But according to the literature data [6] polysilicate ions are also present in the solution. That is why ionization scheme of silicic acid should be completed by two reactions:

$$2HSiO_3^- + H^+ \overset{K_3}{=} Si_2O_5^{2-} + H_2O\,; \log K_3 = 3{,}34$$

$$Si_2O_5^{2-} + H^+ \overset{K_4}{=} = H_2SiO_2O_5 \quad \log K_4 = -9{,}80$$

Silicic acid has a complicate mechanism of ionization in aqueous solutions. That is why it is rather difficult to execute calculations for the determination of the ionization degree of groups and quantity of the free alkali at different pH values [7]. Determination of these parameters is necessary for the calculation of inductive period of gel formation. That is why experimental methods based on potentiometric researches of pH dependence on concentrations of sodium silicates and alkalis and pH change in the result of dilution with water have been used in this work [8-10].

3. Purposes and problems of a research

The possibility of usage of direct and reverse titration of silicate gels for determination of critical pH was researched. pH change of silicate-alkali mixture has been studied while addition of alkalis, salt water and sodium hydrogen carbonate solutions.

4. Materials and methods of a research

Methods of direct and reverse titration were used for the determination of various additives influence of the pH value change of sodium silicate mixtures. Alkali, salt water and solutions of carbonate and hydrogen carbonate of sodium were used as additives.

5. Research results

Typical curve of the direct titration is shown on fig. 1.

Point of equivalency corresponds to the maximum slope of curve. As a rule this point corresponds to the pH ≈7. Alkali's concentration has been calculated using formula:

$$V_{HCl} \cdot N_{HCl} = V_{NaOH} \cdot N_{NaOH}$$

where V_{HCl} – acid's volume used on the titration; N_{HCl} – normality of the acid solution; V_{NaOH} – volume of the alkali solution, which we titrated; N_{NaOH} – normality of the alkali solution.

As s rule, concentration of the hydrochloric acid is equal to 1 mol-eqv/dm^3.

The amount of the free alkali can be calculated using titration curve (fig.1) in the all investigated range.

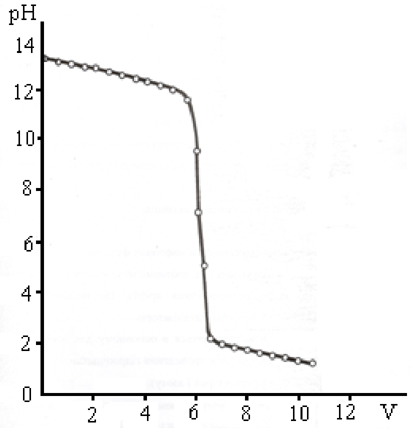

Fig. 1. Typical titration curve.
Titrant is - 1N HCl;
Dilutant is 0.5 % NaCl;
Volume of NaOH is 25 cm^3;
V - volume of HCl, dm^3.

Determination of the equivalence point and concentration of the free alkali is more complicate process for the titration of alkali-silicate solutions.

Curve for the titration of 1% solution of sodium silicate with 1N solution of HCl is shown on fig. 2. These solutions have been prepared on the basis of 0.5 % NaCl solution because of a small change of the ionic power of the solution.

As you see from fig. 2, titration curve is gentler than in case of alkali titration. It is more difficult to find the equivalence point because of possibility of gel formation while titration of silicate solution with an acid. Because of this additional errors can be formed in the calculation of free alkali concentration.

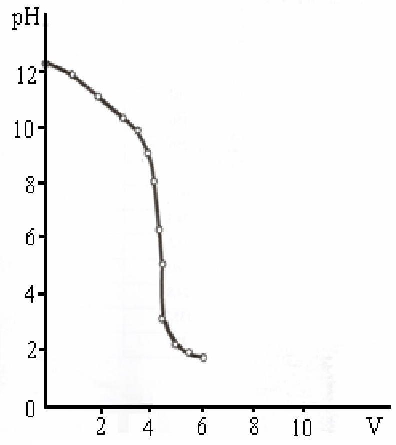

Fig. 2. Direct titration of 1% sodium silicate solution.
V - volume of HCl, cm^3.
Titrant - 1N. HCl.
Dilutant - 0.5 % NaCl.

It is expedient to use method of additives or reverse titration for the determination of the content of free alkali. In this case silicate solutions are titrated with a strong acid to the fixed value of pH and then reverse titration with the alkali has been executed. The example of the reverse titration of 1% sodium silicate solution is shown on fig. 3.

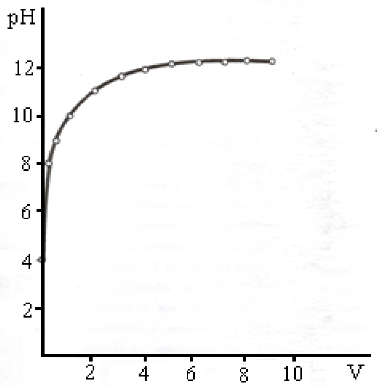

Fig. 3. Reverse titration of 1 % sodium silicate solution.
V - volume of NaOH, cm^3.
Titrant - 1 N. NaOH
Dilutant - 0.5 % NaCl

Method of additives helps to solve one the main problems in the design of technology of silicate action. This problem consists in the calculation the amount of alkali, which is neutralized by ore when silicate solutions have been filtrated in porous medium. Buffer capacity of the certain ore as neutralizator of alkali is the additional parameter, which is necessary for such calculation.

Results of the potentiometric researches of silicate solutions of various concentrations are shown in table 1. These results are also initial data for the calculation of the amount of alkali, which is necessary for the preparation of sodium silicate solution with certain pH values.

Table 1

pH change of silicate solutions at direct titration of HCl and reverse titration of NaOH. Solvent is 0,5% NaCl. The volume of a sample is 25 cm^3. Concentration of titrants is 1N.

1 % silicate solution				3 %- silicate solution				5 % silicate solution			
Direct titration		Reverse titration		Direct titration		Reverse titration		Direct titration		Reverse titration	
Volume of HCl,cm^3	pH	Volume of NaOH,cm^3	pH	Volume of HCl,cm^3	pH	Volume of NaOH,cm^3	pH	Volume of HCl,cm^3	pH	Volume of NaOH, cm^3	pH
0,0	12,30	0,0	4,00	0,0	12,15	0,0	4,90	0,0	12,00	0,0	4,65
1,0	11,90	0,1	8,10	1,0	11,70	0,1	6,50	1,0	11,70	0,1	6,30
2,0	11,00	0,2	8,50	2,0	11,30	0,2	7,00	2,0	11,45	0,2	6,85
2,5	10,65	0,3	9,20	2,5	11,15	0,3	7,40	3,0	11,20	0,3	7,05
3,0	10,25	0,4	9,60	3,0	10,95	0,4	7,70	4,0	10,95	0,4	7,30
3,5	9,90	0,5	9,80	3,5	10,80	0,5	8,00	5,0	10,75	0,5	7,40
4,0	9,00	0,6	10,60	4,0	10,60	0,6	8,25	6,0	10,50	0,6	7,50
4,1	8,50	0,7	10,20	5,09	10,30	0,7	8,45	7,0	10,35	0,7	7,60
4,2	8,00	0,8	10,35	6,0	9,80	0,8	8,70	8,0	10,10	0,8	7,95
4,3	7,55	0,9	10,45	6,9	9,20	0,9	8,90	9,0	9,90	1,0	8,00
4,4	6,30	1,0	10,55	7,0	9,15	1,0	9,15	10,0	9,30	1,5	8,75

4,5	3,10	1,1	10,60	7,4	8,70	1,3	9,50	11,0	8,55	2,0	9,30
4,6	2,80			7,5	8,50	1,5	9,75	11,2	8,40	2,5	9,50
4,7	2,50			7,6	8,30	2,0	10,25	11,3	8,20	3,0	9,70
4,8	2,35			7,7	8,10	2,3	10,45	11,4	8,00	3,5	9,95
4,9	2,25			7,8	7,80	2,5	10,55	11,5	7,85	4,0	10,15
5,0	2,20			7,9	7,50	3,0	10,80	11,6	7,65	4,5	10,35
				8,0	7,10	6,0	11,40	11,7	7,50	5,0	10,60
				8,1	6,60	9,0	12,10	11,8	7,30	5,5	10,70
				8,2	6,00	12,0	12,40	11,9	7,00	6,0	10,80
				8,3	3,50			12,0	6,65	7,0	11,00
				8,4	2,80			12,1	6,30	8,0	11,20
				8,5	2,55			12,2	6,00	9,0	11,35
				8,6	2,40			12,3	5,45	10,0	11,55
				8,7	2,30					11,0	11,75
				8,8	2,20					12,0	11,95
										13,0	12,10

Table 2

pH change of the silicate solution and $NaHCO_3$ solution in the result of titration with HCl and NaOH. Solvent is 0.5 % NaCl +0.1 % $NaHCO_3$ solution. Sample's volume is 25 cm^3.

$NaHCO_3$ solution				1 % sodium silicate solution				3 % sodium silicate solution			
Direct titration		Reverse titration		Direct titration		Reverse titration		Direct titration		Reverse titration	
Volume of HCl, cm^3	pH	Volume of HCl, cm^3	pH	Volume of HCl, cm^3	pH	Volume of NaOH, см^3	pH	Volume of HCl, см^3	pH	Volume of NaOH, см^3	pH
0,0	8,05	0,0	1,60	0,0	10,50	0,0	2,00	0,0	12,60	0,0	2,20
0,1	6,60	0,1	1,70	0,5	10,05	0,5	2,15	1,0	12,65	0,4	2,70
0,2	5,80	2,0	2,00	1,0	9,40	1,0	3,50	2,0	12,25	0,5	3,50
0,3	3,00	2,1	2,10	1,1	9,25	1,1	6,50	3,0	11,85	0,6	6,20
0,4	2,60	2,3	2,20	1,2	9,05	1,2	7,80	4,0	10,95	0,7	7,30
0,5	2,40	2,5	2,40	1,3	8,80	1,3	8,50	5,0	10,05	0,8	8,10
0,6	2,30	2,6	2,55	1,4	8,45	1,4	9,05	5,5	9,65	0,9	8,70
0,7	2,20	2,7	2,85	1,5	7,95	1,5	9,40	5,7	9,40	1,0	9,10
0,8	2,10	2,8	5,20	1,6	7,25	1,6	9,65	5,8	9,25	1,1	9,35
0,9	2,05	2,9	10,40	1,7	6,65	1,7	9,80	5,9	9,05	1,2	9,70
1,0	2,00	3,0	11,15	1,8	6,15	1,8	10,05	6,0	8,85	1,3	9,90
2,0	1,70	3,1	11,40	1,9	5,35	1,9	10,25	6,1	8,60	1,4	10,05
3,0	1,60			2,0	2,90	2,0	10,40	6,2	8,25	1,5	10,15
				2,1	2,55	2,1	10,45	6,3	7,60	1,6	10,35
				2,2	2,40	2,2	10,65	6,4	6,85	1,7	10,50
				2,5	2,15	2,3	10,75	6,5	6,30	1,8	10,60
				3,0	1,95			6,6	5,85	1,9	10,70
								6,7	4,30	2,0	10,80
								6,8	2,80	4,0	11,20
								6,9	2,60	6,0	11,70
								7,0	2,45	7,0	11,90
								7,1	2,30	10,0	12,45
								7,2	2,20		
								7,3	2,15		

pH value change when diluting the mixture with salt water. Influence of hydrogen carbonates and carbonates on the dynamics of pH change.

pH value changes in the result of dilution of strong alkalis by water. The pH value is equal to $pH = -\log[H^+]$, that is why dependence of pH from the logarithm of dilution is linear if there are no adverse reactions. Really, according to experimental data this dependence is close to linear.

There is a deviation from the linear dependence in case of silicates and other salts formed by a strong base and weak acid (especially polyprotic).

pH change in the result of dilution 1% silicate solution with a solvent (0,5 % NaCl solution) is shown on fig. 4-a.

As you see in fig. 4-a there is a small deviation from linearity because of buffer capacity of silicate solutions. Additional deviation from linear dependence of pH from dilution takes place in the solutions, which contain hydrogen carbonate of sodium (fig. 4-b).

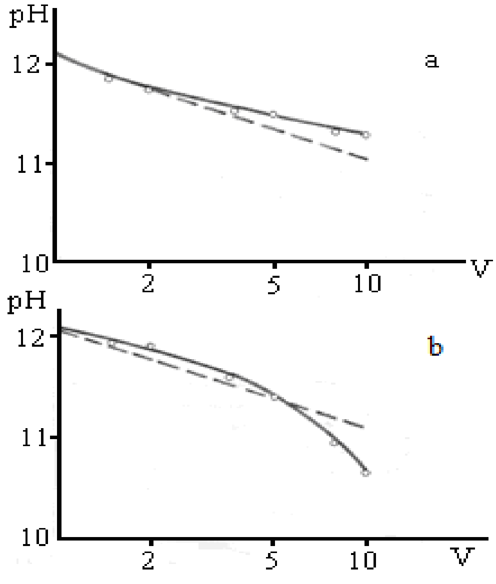

Fig. 4. Change of pH value of 1 % sodium silicate solution when diluting.
a - dilutant - 0.5 % NaCl;
b - dilutant 0.5 % NaCl + 0.1 % $NaHCO_3$
V - volume of the dilutant, cm^3.
---- theoretical curve for NaOH

Hydrogen carbonates and carbonates, as a rule, are present in most surface and underground water, including salt water and seam water of oil reservoirs. Hydrogen carbonates are salts of weak carbonate acid and have the same buffer capacity as silicates unlike chlorides and sulfates, which are components of natural water. The high pH value of salt water is caused by the presence of hydrogen carbonates.

Results of the direct and reverse titration of silicate solutions prepared using water, which contains 1 g/l of $NaHCO_3$, are shown in table 2. As it can be seen from a comparison of data, given in tables 1and 2, the presence of hydrogen carbonates in such small amount affects on the position of a titration curve of silicates. Such phenomenon can be explained by the value of concentration of silicate. It is higher than concentration of hydrogen carbonates at least in one order.

Calculations show, that 1 g/l of $NaHCO_3$ corresponds to approximately 0.16 g/l of NaOH. Seam water contains smaller amount of hydrogen carbonates and salt water also contains approximately in 5 times smaller amount of these compounds. That is why hydrogen carbonates practically do not impact on pH value of silicate solutions, which are mixed with seam and salt water.

But it is necessary to take into account the influence of hydrogen carbonates when executing accurate calculations in the design of technology of silicate usage.

Dilution of silicate-alkali solutions with salt water or its model is accompanied with faster decreasing of pH of the solution than in case of usage of water without salts of hardness. It happens because not only dilution process takes place, but the precipitation of silicates and hydroxides of calcium and magnesium proceeds. Fig. 5 shows the dependence of pH value on the degree of dilution with salt water at different pH values of the initial solution. As you can see from fig. 5 the sharp decreasing of the pH value happens after the dilution in two times.

Potentiometric analysis data gives a possibility to calculate the amount of silicate, which precipitates, on the basis of comparison of dependence of pH value on the degree of dilution of salt water and its model, which does not contain salts of hardness.

Curves of pH dependence of 5 % sodium silicate solutions from the degree of dilution by salt water at 25 °C and 60 °C are shown on fig. 6. We can say on the result of the experimental data, that increase of temperature is slightly impact on pH change of solutions and dependence curves are practically identical.

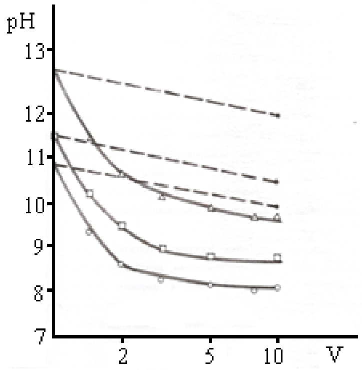

Fig. 5. pH change of the sodium silikate solution when diluting with salt water.
V- volume of dilutant, cm^3.
Dilutant - 0.05 % NaOH.
Temperature 25 ^{0}C.
---- theoretical curve for NaOH.

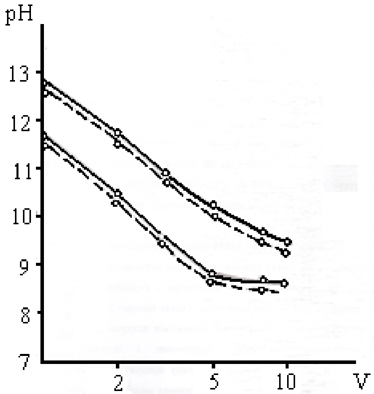

Fig. 6. Change of pH value of sodium silicate solution when dilting with salt water at different temperatures.
Dilutant - 0.05 % NaOH.
——— temperature 25 ^{0}C.
– – – temperature 60 ^{0}C.

The same dependences have been obtained for 2 % and 3 % sodium silicate solutions. There are the same regularities when diluting solutions of salt water with certain mineralization.

6. Discussion of results

Alkali-silicate mixtures would be solutions for a long time when filtration proceeds in the porous medium at pH > pH_{crit}. The time of such solutions existence must be characterized by a before inductive period. When reaching the critical pH value, a gel is formed during an inductive period or the time of gel formation. As a rule, the before inductive period is in ten times or even more greater than the time of gel formation

7. Conclusions

1. Methods of additives or reverse titration of a solution with an alkali are more accurate for the determination of the free alkali content in silicate solutions.
2. Dilution of silicate-alkali solutions with salt water or its model is accompanied by faster decrease of pH value than in case of water without hardness salts.
3. The influence of hydrogen carbonates should be taken into account when executing accurate calculations, which are used in the design of usage technology of silicate-alkali mixtures.

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Визначення критичної величини рН гелеутворення силікатно-лужних композицій різного складу

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Мета: Метою цієї статті є дослідження можливості застосування прямого та зворотного титрування силікатних гелів для визначення критичного рН. Проаналізовано зміну рН силікатно-лужної композиції при додаванні лугів, морської води та розчинів гідрокарбонату натрію. **Методи дослідження:** Для визначення впливу різноманітних добавок на результати зміни рН композицій силікату натрію використовували методи прямого та зворотного титрування. В якості добавок застосовували луг, морську воду та розчини карбонату і гідрокарбонату натрію. **Результати:** Для визначення вмісту вільного лугу в силікатних розчинах більш відповідним є метод добавок або зворотне титрування розчину лугом. Розбавлення силікатно-лужних розчинів морською водою або її моделлю супроводжується більш швидкім зниженням рН розчину, ніж у випадку використання вод, які не містять солей твердості. При проведенні точних розрахунків, які використовують при розробці технології застосування силікатів-лужних композицій, повинен враховуватися вплив гідрокарбонатів. **Обговорення:** При фільтрації лужно-силікатних композицій в пористому середовищі при значеннях $pH > pH_{крит.}$, вони залишатимуться у вигляді розчинів достатньо довго. Час існування таких розчинів повинен характеризуватися передіндукційним періодом. Досягши критичного значення рН формуватиметься гель протягом індукційного періоду або часу гелеутворення. Як правило, передіндукційний період на порядок або більше перевищує час гелеутворення.

Ключові слова: величина рН; гелеутворення; динаміка зміни рН; силікат натрію.

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Определение критической величины рН гелеобразования силикатно-щелочных композиций разного состава

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Цель: Целью этой статьи является исследование возможности применения прямого и обратного титрования силикатных гелей для определения критического pH. Проанализировано изменение pH силикатно-щелочной композиции при добавлении щелочей, морской воды и растворов гидрокарбоната натрия. **Методы исследования:** Для определения влияния различных добавок на результаты изменения pH композиций силиката натрия использовали методы прямого и обратного титрования. В качестве добавок применяли щелочь, морскую воду и растворы карбоната и гидрокарбоната натрия. **Результаты:** Для определения содержания свободной щелочи в силикатных растворах более подходящим является метод добавок или обратное титрование раствора щелочи. Разбавление силикатно-щелочных растворов морской водой или ее моделью сопровождается более быстрым снижением pH раствора, чем в случае использования вод, не содержащих солей жесткости. При проведении точных расчетов, используемых при разработке технологии применения силикатно-щелочных композиций, должно учитываться влияние гидрокарбонатов. **Обсуждение:** При фильтрации щелочно-силикатных композиций в пористой среде при значениях $pH > pH_{крит.}$, они будут оставаться в виде растворов достаточно долго. Время существования таких растворов должно характеризоваться перединдукционным периодом. При достижении критического значения pH формируется гель в течение индукционного периода или времени гелеобразования. Как правило, перединдукционный период на порядок или более превышает время гелеобразования.

Ключевые слова: величина pH; гелеобразование; динамика изменения pH; силикат натрия.

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