A STUDY OF SOME PROPERTIES OF SILICIC ACID GELS

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INTRODUCTION

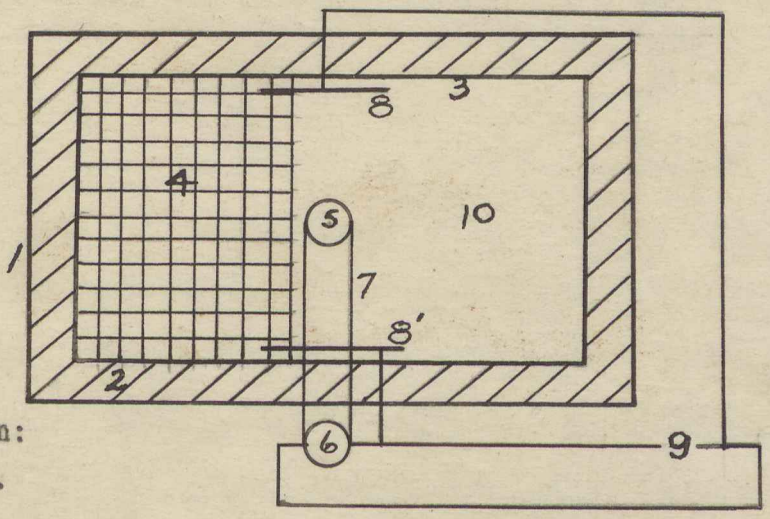
When a solution of sodium silicate or water glass is mixed with an acid sooner or later a gel is formed. The speed of gelation is known to be dependent upon (1) the concentration of water glass, (2) the excess of acid or alkali present, (3) agitation, (4) temperature, (5) and the kind of acid used. In my work with the setting of the gels these five points were kept as constant as possible. I investigated the effect of the formation of the sodium acetate salt, which is one of the products of the reaction when sodium silicate and acetic acid are mixed. An addition of the two common ions was a means of determining this effect. Another line of investigation was followed with the addition of one of the common ions, namely sodium, by means of sodium chloride, which gave entirely different results.

HISTORICAL

Flemming,[1] in the year of 1902, studied the problem of the setting of silicic acid gels as a function of the temperature and the concentration of the acid. Flemming considered the time of set to depend wholly upon the concentration of silicic acid liberated. H.N.Holmes,[2] as well as H.A.Fells[3] and J.B.Firth,[4] and later H.A.Letteron[5] continued their investigations of the setting of the gels in the same manner as Flemming.

APPARATUS

Since it was known that any change in temperature had a very marked effect on the time of setting of the gels, a thermostat which could be kept and regulated at the desired temperature was necessary. I constructed a thermostat out of a small rectangular glass tank 40 cm. x 25 cm. x 30 cm. This was placed in a large wooden box and sawdust, which was used as an insulating material, was packed around it. The water was stirred by paddles attached to a shaft which was operated by a small motor. The water was heated by means of two sheet copper electrodes 5 cm. square placed in the tank. An approximate calculation of the heating up of the water by electrical conduction showed that it took 15 minutes to raise the temperature 1° Centigrade. With this thermostat the temperature could be held as close as $\pm .1^{\circ}$C. The following is a top view of the bath.



Key to diagram:

1. Wooden box.
2. Sawdust.
3. Glass Tank.
4. Screen to hold beakers on in water.
5. Pulley on end of shaft on which are attached paddles.
6. Motor.
7. String
8 - 8'. Copper Electrodes.
9. Source of Electrical Energy.
10. Compartment in bath for graduated flasks.

The water glass and acetic acid were kept in one side of the bath in graduated flasks and the other side was used to keep the beakers containing the measured quantities of water glass and acetic acid.

The acetic acid which I used was glacial acetic acid 99.5%. This was diluted to the desired normality. The water glass used was commercial sodium silicate with the formula $Na_2O\ 3\ SiO_2$

I found that a specific gravity of 1.104 at 20°C. for the water glass formed, with the acetic acid at desired proportions, a gel in a reasonable length of time. The sodium acetate and sodium chloride solutions which I used were both made up as .5 N.

All measurements of quantities were made either with pipettes or with burettes since it was observed that it was very essential to get the exact proportions for a good agreement of different readings for the time of set of the gels.

In making the gels the water glass was placed in the beaker and the other constituents, namely the acid, water, sodium acetate or sodium chloride, were placed in another beaker. Both of these were put in the bath until the desired temperature was reached. In mixing the two, it was my custom to pour the water glass into the acid solution. The gel was considered to have set when a uniform glass rod of approximately 3 mm. in diameter and 8 cm. in length would stand erect in the gel. It was thought at first that the repeated operation of placing the rod in and out of the gel to test its setting would injure the structure of the gel. This was found not to be true. It was a very accurate way of determining the time of set, since I could

measure the set as accurate as ± 1 second.

RESULTS

I first tried an experiment to determine the conditions for the quickest set of the gel with only water glass and acid. I used as an indicator phenolphtalein. Table I gives the results which I obtained for the time of setting at constant temperature.

I next tried the effect of the addition of sodium acetate in the mixture keeping the amount of water glass and acid constant but varying the amount of water and sodium acetate. Table II shows these results. Figure 1 indicates the results as time plotted against the amount of sodium acetate added.

The effect of the addition of sodium chloride to the acid and water solution on the time of set is shown in table III.

DISCUSSION RESULTS

The results obtained in determining the time of quickest set show that a very slight alkalinity in the neighborhood of the neutralization point brings about this condition. It seems, therefore, that there must be a catalytic effect of the hydrogen or hydroxyl ions in the setting of the gels. I was hoping to find the effect of the hydrogen ion concentration on the setting of the gel, but the time was too limited.

The results obtained for the effect of the addition of sodium acetate seem to indicate that there is a minimum point at which the quickest set occurs. This is a very interesting result. If there is a catalytic effect of the hydrogen ion

concentration, the excess of free acetic acid must be greatly repressed in ionization by the sodium acetate. However, we would not expect a minimum point if such were the case. It seems to me such an effect would be a steady increase or a gradual decrease of the time of setting.

The results obtained when sodium chloride was used showed that there was no appreciable effect in the time of setting. This eliminates the possibility of the sodium chloride having a very marked effect on the activity of the acetic acid and thus affecting the time of set.

SUMMARY

The quickest set of the gel occurs when the solution is almost neutral. It is just slightly alkaline.

The effect of the sodium acetate seems to indicate a minimum effect in the time of setting.

The sodium chloride seems to have a very small effect on the activity of the acetic acid.

BIBLIOGRAPHY

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2 - H.N.Holmes	J.Phys.Chem. 22,410	(1918)
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TABLE I

IN. c.c. CH_3COOH	WATER c.c.	GLASS	TIME	TEMP.	COLOR OF INDICATOR
11	25		85	22.5°C	Red
11.2	25		50	22.5°C	Red
11.5	25		45	22.5°C	Deep Pink
11.8	25		42	22.5°C	Pink
12.1	25		40	22.5°C	Light Pink
12.4	25		130	22.5°C	Colorless

TABLE II

c.c. W.G.	c.c. 2N CH_3COOH	c.c. H_2O	c.c. .5N NA $C_2H_3O_2$	TIME	TEMP.
50	25	25	0	166 Sec.	27°C
50	25	23	2	156 "	27°C
50	25	21	4	145 "	27°C
50	25	19	6	142 "	27°C
50	25	17	8	142 "	27°C
50	25	15	10	132 "	27°C
50	25	13	12	136 "	27°C
50	25	11	14	140 "	27°C
50	25	9	16	140 "	27°C
50	25	7	18	145 "	27°C
50	25	5	20	146 "	27°C
50	25	3	22	145 "	27°C
50	25	0	25	149 "	27°C

TABLE III

c.c. W.G.	c.c. 2N CH_3COOH	c.c. H_2O	c.c. .5N NaCl	TIME	TEMP.
50	25	25	0	164 Sec.	27°C
50	25	23	2	163 "	27
50	25	21	4	160 "	27
50	25	19	6	161 "	27
50	25	17	8	159 "	27
50	25	15	10	162 "	27
50	25	13	12	163 "	27
50	25	11	14	158 "	27
50	25	9	16	163 "	27
50	25	7	18	161 "	27
50	25	5	20	162 "	27
50	25	3	22	164 "	27
50	25	0	25	165 "	27