INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO F.W. Kanzler AT Burlington DATE August 18, 1975

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SUBJECT Evaluation of Deionized Gelatin

Four samples of deionized gelatin were evaluated in the Analytical Laboratory. The samples were studied for pH and were also evaluated using the bottle polymerizer.

For the purposes of this report, the following notation will be used:

"A" - current type "A" now being used
"B" - current type "B" now being used
"A^1" - deionized type "A" gelatin, lot 512510A
"A^2" - deionized type "A" gelatin, lot 7514402
"B^1" - deionized type "B" gelatin, lot 7503902
"B^2" - deionized type "B" gelatin, lot K515902B

Summary

The pH studies of the deionized gelatin indicate the pH of the deionized "A" type gelatin solutions will have a pH before polymerization of about .3 to .6 pH units higher than the "A" type gelatin currently being used. The deionized "A"/"B" combinations also are higher than the current "A"/"B" type being used, about .1 to .2 pH units. Bottle polymerization studies indicate the deionized "A" type to be more reliable in avoiding set-ups than the current "A" type now being used. None of the bottles using the deionized "A" type set up while one of the bottles using the currently-used "A" type was bb's. All the "A"/"B" systems yielded good resin. Screen analyses of the resin shows the current "A"/"B" type resins yielded a finer resin in the bottle polymerization than the deionized gelatin. Relative viscosity measurements showed no significant differences between the gelatins currently being used and the deionized type.

I. pH Studies

The following formulation was used for this study:

demineralized water	- 325.2 gms	
L_2O_2	- 0.26 gms	
total gelatin	- 0.66 gms	temperature - 135°F ±5°
TCE	- 1.78 gms	
MVAc	- 33.4 gms	

The gelatin used for each study is the type specified. Where two gelatins were used, 0.33 gms of each type were used. The same raw materials were used for all of these studies.

A. Gelatin "A^1"

The initial pH of this solution was about 0.6 pH units higher than the previous studies made using gelatin "A". The pH prior to the

addition of TCE was about 0.3 units higher than the previous studies. After the addition of TCE, the pH difference is only about 0.2 units. Five minutes after the addition of MVAc, the pH differences is only about 0.1 units. This seems to indicate that the pH differences of the final mixture prior to the addition of VCM are quite small.

B. Gelatin "A^2"

This study followed the same patterns as Study I except the pH"s ran about 0.1 pH units higher. So the differences are 0.1 units greater than those recorded above. The pH difference of the solution prior to addition of VCM is 0.2 units. In earlier studies of type "A" gelatin using a slightly different formulation, a final difference of about 0.1 units was found on duplicate runs. These results will give slightly higher pH's than the type "A" now being used.

C. "A^1"/"B^1" Gelatin

The pH's levels in this study ran about 0.4 higher initially than type "A^1". The final pH is about 0.2 pH units higher than the study for type "A^1".

D. "A^2"/"B^1" Gelatin

This study follows a similar pattern to "A^1"/"B^1". The pH's are less than 0.1 units higher than "A^1"/"B^1".

E. "A^2"/"B^1" Gelatin

The study is similar to the two above, however, the pH's run about 0.1 pH units higher than "A^1"/"B^1".

F. "A^2"/"B^2" Gelatin

This study is similar to the three above while the pH's are less than "A^1"/"B^2".

These studies show that the "A"/"B" combinations have pH's about 0.3 to 0.4 higher than deionized "A" used by itself. Also, type "A^2" when used in combinations with "B^1"/"B^2" yield a pH of about 0.1 units higher. This follows the same patterns as the studies above, where "A^2" was slightly higher in final pH than "A^1". "B^2" yields a final ph about 0.5 units higher than "B^1" in combinations with "A^1" or "A^2".

II. Bottle Polymerization Studies

Three bottle polymerization runs of six bottles each were run:

Run	Bottle	Gelatin Type	Result
1	1	"A"	bb's
	2	"A"	resin
	3	"A^1"	resin
	4	"A^1"	resin
	5	"A^2"	resin
	6	"A^2"	resin

Run	Bottle	Gelatin Type	Result
2	1	"A"/"B"	resin
	2	"A"/"B"	resin
	3	"A^1"/"B^1"	resin
	4	"A^1"/"B^1"	resin
	5	"A^1"/"B^2"	resin
	6	"A^1"/"B^2"	resin
3	1	"A"/"B"	resin
	2	"A"/"B"	resin
	3	"A^2"/"B^1"	resin
	4	"A^2"/"B^1"	resin
	5	"A^2"/"B^2"	resin
	6	"A^2"/"B^2"	resin

This study seems to indicate that the deionized gelatins are more reliable than the current "A" type. In study I, all of the deionized gelatin bottles had resin, half the "A" type currently used made bb's. Also, the "A"/"B" combination bottles made resin in all twelve bottles run.

The bottles all had gelatinous spherical particles of resin which floated. These are thought to be soft spheres of resin with monomer trapped inside. These spheres appeared to be most abundant in bottles made using type "B^2" gelatin.

The resin, when dried, showed white fibrous particles of resin and also hard glassy spheres of resin.

These runs show the reliability of an "A"/"B" suspension system versus the "A" type by itself. None of the "A"/"B" bottles set up.

The pH's of the mother liquors of the "A "/"B" run were measured and are on Table II. These results indicate the range of pH's over which this system gives colloidial protection.

Screen analysis of resin from Polymerization

The copolymer from the seventeen bottles which yielded "good" resin was analyzed for particle size distribution. The results are tabulated on Table III. The screen analysis was done using 50 gms of resin and 2.5 gms of graphite.

The results indicate that the "A"/"B" type gelatin now in use gives a finer resin than the deionized types. This is most evident in the bottles run with type "A^2" by itself or in combination with "B^1" or "B^2". None of the bottles made with type "A^2" had resin finer than 200 mesh. The presence of a resin smaller than 200 mesh in the bottles using type "A^1" by itself or in combination with "B^1" or "B^2" indicates that this type is a candidate for further evaluation. Only one bottle, an "A^1"/"B^2" combination, showed no resin finer than 200 mesh. It is felt that a Pilot Plant run would give a better indication as to actual plant behavior.

Relative Viscosity of Bottle Resin

The relative viscosity of the resin made in the bottle polymerization was measured. With the exception of bottles 2 - 3 (relative viscosity of 1.51),

all the results are in the range of 1.56 to 1.60. The viscosities show no dramatic changes from gelatin type to gelatin type. This indicates that in bottle polymerization, the types of gelatin used in this study will have no great effect upon the relative viscosity and hence, the molecular weight.

The screen and viscosity data should be considered, however, with caution. The writer was able to find no other screen or viscosity data for copolymer made in bottles. Therefore, it is not known if any of the values found are typical or "fluxes". It is also not known what the correlation is between the physical properties of bottle resin and plant resin.

If a bench scale reactor had been available, the writer feels that the data would be more meaningful. A bench scale reactor would better duplicate actual plant conditions which would give a better picture of what would happen in the plant. If studies similar to the one described in this report are planned in the future, such equipment should be acquired.

E. Young

EY/gs

Attachments (9)

TABLE 1 - 1

pH STUDY USING GELATIN "A[1]"

Addition of L_2O_2 and Gelatin "A[1]"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	4.90	1	4.90	1	4.75
2	4.90	2	4.90	2	4.72
3	4.90	3	4.89	3	4.70
4	4.90	4	4.88	4	4.69
5	4.90	5	4.88	5	4.66
6	4.89	6	4.85		
7	4.90	7	4.83		
8	4.90	8	4.82		
9	4.90	9	4.81		
10	4.90	10	4.80		
11	4.90				
12	4.90				
13	4.90				
14	4.90				
15	4.90				
16	4.90				
17	4.90				
18	4.90				
19	4.90				
20	4.90				

TABLE 1 - 2

pH STUDY USING GELATIN "A^2"

Addition of L_2O_2 and Gelatin "A^2"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	4.95	1	5.00	1	4.80
2	5.00	2	4.99	2	4.81
3	5.01	3	4.98	3	4.80
4	5.00	4	4.95	4	4.80
5	5.00	5	4.92	5	4.78
6	5.00	6	4.91		
7	5.00	7	4.90		
8	5.00	8	4.89		
9	5.00	9	4.88		
10	5.00	10	4.88		
11	5.00				
12	5.00				
13	5.00				
14	5.00				
15	5.00				
16	5.00				
17	5.00				
18	5.00				
19	5.00				
20	5.00				

TABLE 1 - 3

pH STUDY USING GELATIN "A[1]" AND "B[1]"

Addition of L_2O_2 and Gelatin "A[1] and "B[1]"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	5.22	1	5.30	1	5.10
2	5.30	2	5.30	2	5.02
3	5.30	3	5.30	3	5.00
4	5.30	4	5.29	4	4.98
5	5.30	5	5.29	5	4.92
6	5.30	6	5.28		
7	5.29	7	5.26		
8	5.29	8	5.24		
9	5.29	9	5.22		
10	5.29	10	5.22		
11	5.29				
12	5.29				
13	5.29				
14	5.29				
15	5.29				
16	5.30				
17	5.29				
18	5.29				
19	5.29				
20	5.30				

TABLE 1 - 4

pH STUDY USING GELATIN "A[1]" and "B[2]"

Addition of L_2O_2 and Gelatin "A[1]" and "B[2]"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	5.32	1	5.32	1	5.10
2	5.38	2	5.33	2	5.09
3	5.38	3	5.35	3	5.05
4	5.36	4	5.32	4	5.00
5	5.35	5	5.32	5	4.99
6	5.37	6	5.31		
7	5.35	7	5.30		
8	5.34	8	5.30		
9	5.33	9	5.30		
10	5.33	10	5.30		
11	5.33				
12	5.35				
13	5.33				
14	5.32				
15	5.32				
16	5.32				
17	5.32				
18	5.32				
19	5.33				
20	5.34				

TABLE 1 - 5

pH STUDY USING GELATIN "A[2]" AND "B[1]"

Addition of L_2O_2 and Gelatin "A[2]" and "B[1]"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	5.43	1	5.48	1	5.18
2	5.48	2	5.46	2	5.14
3	5.48	3	5.45	3	5.10
4	5.48	4	5.43	4	5.06
5	5.48	5	5.42	5	5.20
6	5.48	6	5.41		
7	5.48	7	5.41		
8	5.46	8	5.40		
9	5.48	9	5.40		
10	5.48	10	5.40		
11	5.48				
12	5.48				
13	5.48				
14	5.46				
15	5.48				
16	5.45				
17	5.45				
18	5.45				
19	5.44				
20	5.45				

TABLE 1 - 6

pH STUDY USING GELATIN "A^2" AND "B^2"

Addition of L_2O_2 and Gelatin "A^2" and "B^2"		Addition of TCE		Addition of MVAc	
Min	pH	Min	pH	Min	pH
1	5.60	1	5.52	1	5.23
2	5.59	2	5.52	2	5.18
3	5.55	3	5.51	3	5.13
4	5.52	4	5.50	4	5.09
5	5.52	5	5.50	5	5.05
6	5.52	6	5.50		
7	5.52	7	5.49		
8	5.52	8	5.48		
9	5.52	9	5.48		
10	5.51	10	5.46		
11	5.52				
12	5.51				
13	5.52				
14	5.51				
15	5.50				
16	5.51				
17	5.52				
18	5.51				
19	5.51				
20	5.51				

TABLE 2

pH OF MOTHER LIQUOR IN BOTTLES FORM RUNS 2 & 3

BOTTLE	GELATIN	pH
2-1	"A" & "B"	3.78
2-2	"A" & "B"	3.65
2-3	"A^1" & "B^1"	3.96
2-4	"A^1" & "B^1"	4.20
2-5	"A^1" & "B^2"	3.62
2-6	"A^1" & "B^2"	3.92
3-1	"A" & "B"	4.01
3-2	"A" & "B"	3.95
3-3	"A^2" & "B^1"	4.20
3-4	"A^2" & "B^1"	3.98
3-5	"A^2" & "B^2"	3.80
3-6	"A^2" & "B^2"	3.98

TABLE 3

SCREEN ANALYSIS

Bottle	Gelatin	Mesh 35	60	80	100	140	200	Thru
1-1	"A"	6	18	48	64	76	96	4
1-2	"A"	Bee Bees						
1-3	"A^1"	12	34	56	68	80	96	4
1-4	"A^1"	4	22	56	66	72	98	2
1-5	"A^2"	6	34	64	76	86	100	0
1-6	"A^2"	10	40	72	84	94	100	0
2-1	"A" & "B"	6	24	46	58	62	90	10
2-2	"A" & "B"	26	42	56	66	74	94	6
2-3	"A^1" & "B^1"	20	44	64	78	84	96	4
2-4	"A^1" & "B^1"	10	26	56	70	76	96	4
2-5	"A^1" & "B^2"	2	34	68	80	84	100	0
2-6	"A^1" & "B^2"	4	34	68	80	82	96	4
3-1	"A" & "B"	4	16	36	50	62	90	10
3-2	"A" & "B"	2	10	32	48	78	96	4
3-3	"A^2" & "B^1"	8	32	66	78	88	100	0
3-4	"A^2" & "B^1"	6	32	64	82	86	100	0
3-5	"A^2" & "B^2"	4	40	54	86	90	100	0
3-6	"A^2" & "B^2"	6	36	66	78	80	100	0

TABLE 4

RELATIVE VISCOSITY

Sample	Viscosity
1-1	1.59
1-2	Bee Bee's
1-3	1.60
1-4	1.59
1-5	1.59
1-6	1.57
2-1	1.58
2-2	1.57
2-3	1.51
2-4	1.58
2-5	1.57
2-6	1.59
3-1	1.57
3-2	1.58
3-3	1.57
3-4	1.56
3-5	1.58
3-6	1.58