

## *Coagulation of Hevea Latex with Surfactant and Salt II. Batch-Wise Commercial Processing*

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*The feasibility of coagulating fresh or ammoniated latices using di-octylsodium sulphosuccinate and calcium chloride, reported earlier, can be extended to factories already equipped for processing latex in batch-wise coagulating tanks. Cost of the coagulants and technological properties of the resulting rubber compare favourably with acid coagulation.*

It has been reported earlier (JOHN, 1971) that *Hevea* latex can be coagulated within 3 min in the presence of a surfactant (di-octylsodium sulphosuccinate) and a salt (calcium chloride) – termed S/S coagulation. The near-instant gelling makes possible continuous coagulation of a constant feed of latex from bulk storage. The feasibility of such an automated coagulation process has been demonstrated by a pilot-scale operation (JOHN AND NEWSAM, 1969).

Advantages of this process are that the coagulum can be directly fed into the subsequent processing stage, with a saving in labour costs, and that the use of coagulation tank can be dispensed with, resulting in substantial cost savings as well as economy of space. Further, technological properties of the resulting rubber show improvements over the conventional acid-coagulated rubber, especially in respect of nitrogen content, resilience and heat build-up. On the other hand, disadvantages are that latex has to be stored in bulk using a preservative which does not adversely affect subsequent coagulation. Studies were therefore carried out to find out if S/S coagulation can be conducted in batch-wise conventional tanks.

### MATERIALS AND METHODS

#### *Latex*

Fresh latex was obtained from factory bulk. Ammoniated latex was prepared by

adding the required level of ammonia to fresh latex, based on weight of latex; unless otherwise mentioned it contained 0.1% ammonia. Latices with various levels of dry rubber content (d.r.c.) 35, 30, 25, 20 and 15% were prepared by diluting a high d.r.c. latex with water.

#### *Coagulants*

The coagulants used were an anionic surfactant, a salt and formic acid. The surfactant was Aerosol GPG (a 75% solution of di-octylsodium sulphosuccinate in a water-alcohol mixture) and the salt was the commercial grade of calcium chloride, both additions being based on d.r.c. of latex. The surfactant was added as 10%, the salt 5% and formic acid as 2% solutions in water.

#### *Coagulation of Latex*

Both fresh and ammoniated latices were coagulated with formic acid (at pH 5.2) or surfactant and salt, or salt alone. In the case of the latter two, it was sometimes necessary to neutralise part of the ammonia in the ammoniated latex beforehand with formic acid, the extent of neutralisation depending upon the period for which the coagulum was allowed to mature before processing. When both surfactant and salt were used in the same coagulation system,

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the surfactant was added first, followed by the salt, with adequate stirring between the two additions. For producing constant viscosity (CV) rubber, the latex was treated with 0.15% hydroxylamine hydrochloride (HH) on d.r.c. (SEKHAR, 1960) before adding the coagulants.

#### *Processing of Coagulum*

Coagula were processed 8 or 20 h after addition of coagulants by crumbling (SEKHAR *et al.*, 1965). The resulting crumbs were dried at 60°C for two or three days.

#### *Analytical Testing*

Ash, nitrogen, calcium, copper, manganese and acetone extract contents of dry rubber were tested according to the methods described by CHIN (1970).

#### *Technological Testing*

Technological testings were carried out using rubber obtained from 10-gal trials. Vulcanisate properties were tested using ACS 1 and HAF mixes. ACS 1 mix consisted of 100 parts natural rubber, 6 parts zinc oxide, 0.5 part stearic acid, 0.5 part MBT and 3.5 parts sulphur. HAF mix consisted of 100 parts natural rubber, 4 parts zinc oxide, 1.5 parts stearic acid, 45 parts HAF, 5 parts Durex R, and 1.5 parts Nonox ZA.

### RESULTS

#### *Coagulation of Unammoniated Latex*

Unammoniated latex was coagulated with a range of surfactant (0.01–0.1%) and salt (0.025–0.2%) to find out the optimum concentration of the coagulants to fully coagulate the latex in 8 or 20 hours. A parallel experiment was carried out with latex treated with 0.15% HH. Latex was completely coagulated in 8 h in the combined presence of 0.05% surfactant and 0.1% salt, and for latex with HH the quantity of salt

TABLE 1. OPTIMUM CONCENTRATIONS OF COAGULANTS FOR UNAMMONIATED AND AMMONIATED LATICES

Ammonia (%)	HH (%)	Coagulation time (h)	Coagulants	
			Surfactant (%)	Salt (%)
Nil	Nil	8	0.05	0.10
		20	0.01	0.10
		20	—	0.15
Nil	0.15	8	0.05	0.05
		8	0.01	0.20
		20	0.01	0.01
		20	—	0.15
0.1	Nil	8	0.1	0.1
		20	0.07	0.1
0.1	0.15	8	0.1	0.1
		20	0.1	0.1

could be halved to 0.05% or, the surfactant can be reduced to 0.01%, if the salt content is increased to 0.2% (Table 1). When the coagulation time was extended to 20 h, 0.01% surfactant and 0.1% salt, or alternatively 0.15% salt alone, were adequate for both types of latices.

#### *Coagulation of Ammoniated Latex*

Latex ammoniated up to pH 8.5 was completely coagulated in 8 h with 0.1% each of surfactant and salt. At a higher pH, coagulation was incomplete in 8 h even with further addition of coagulants. In 20 h latex ammoniated up to pH 9.0 was completely coagulated with 0.07% surfactant and 0.1% salt. To get complete coagulation with HH the pH had to be reduced to 7.5 prior to the addition of the coagulants (Table 1).

#### *Rubber Content of Latex*

To find out the effect of S/S coagulation on latices with different levels of d.r.c.

which normally varies from 25% to 40%, a series of experiments was carried out using unammoniated and ammoniated latices with and without HH.

Effective coagulation was obtained down to 25% d.r.c. both at 8 and 20 h using the optimum concentrations of surfactant and salt noted earlier. The presence of HH somewhat enhanced coagulation efficiency, complete coagulation being obtained even down to 20% d.r.c. latex.

#### *Effect of Salt on Raw Rubber Properties*

The effect of salt on some of the raw rubber properties was investigated by coagulating unammoniated latex with various levels of salt, processing the coagula after 20 h and testing the resulting rubber for ash, nitrogen and calcium (*Table 2*). It was shown that up to 0.2% salt hardly affected the ash and nitrogen contents, despite the marked increase in the residual calcium content from 14 to 218 p.p.m. In further experiments, even 0.5% salt did not give any increase in ash content; the ash content of rubber coagulated with 0.5% salt was only 0.37%, compared to 0.36% of the acid-coagulated rubber.

TABLE 2. ASH, NITROGEN AND CALCIUM CONTENTS OF RUBBER COAGULATED WITH VARIOUS LEVELS OF SALT

Coagulation system	Salt (%)	Ash (%)	Nitrogen (%)	Calcium (p.p.m.)
Auto Acid	—	0.26	0.42	14
	—	0.25	0.44	13
Salt	0.025	0.27	0.41	29
	0.05	0.26	0.43	46
	0.075	0.27	0.42	88
	0.10	0.27	0.39	117
	0.15	0.29	0.40	137
	0.20	0.29	0.38	218

#### *Technological Properties*

There was no difference in the dirt, copper and manganese contents among the various treatments but only marginal differences in ash content, volatile matter and PRI (*Table 3*). The nitrogen content was uniformly lower, but the acetone extract content was uniformly higher in the S/S coagulated rubbers. The vulcanisate properties of the rubbers obtained from the various S/S coagulation systems were satisfactory (*Table 4*).

#### *Cost of Coagulants*

The cost of coagulating unammoniated latex in 8 h with the S/S system is about the same as that of acid coagulation, but the former is much cheaper when a 20-h

TABLE 3. RAW RUBBER PROPERTIES OF RUBBER FROM S/S AND ACID COAGULATIONS

Property	8 h Coagulation				20 h Coagulation			
	5L		CV		5L		CV	
	S/S (a)	Acid	S/S (b)	Acid	S/S (c)	Acid	S/S (d)	Acid
Ash	0.22	0.20	0.23	0.23	0.18	0.20	0.22	0.22
Nitrogen (%)	0.41	0.42	0.35	0.40	0.38	0.41	0.37	0.42
Volatile matter (%)	0.25	0.24	0.25	0.25	0.22	0.17	0.22	0.20
Acetone extract (%)	3.15	2.09	3.22	3.05	3.03	2.89	3.76	2.80
PRI	92	94	90	87	90	94	90	91

(a) surfactant 0.05% + salt 0.1%

(b) surfactant 0.05% + salt 0.05%

(c) and (d) surfactant 0.01% + salt 0.1%

TABL 4. VULCANISATE PROPERTIES OF RUBBERS FROM S/S AND ACID COAGULATIONS

Property	8 h coagulation				20 h coagulation			
	5L		CV		5L		CV	
	S/S	Acid	S/S	Acid	S/S	Acid	S/S	Acid
<b>ACS 1 Mix</b>								
Cure time at 140°C (min)	40	40	40	40	40	40	40	40
Tensile strength (kg/cm <sup>2</sup> )	221	192	187	170	191	153	197	172
Elongation at break (%)	835	775	790	800	800	770	820	810
Modulus M 600 (kg/cm <sup>2</sup> )	55	64	53	58	54	53	52	45
Relaxed modulus	5.8	5.7	5.6	5.7	5.8	5.5	5.7	5.4
Resilience (%)	83.4	83.0	83.8	82.2	83.9	81.4	83.4	81.3
Mooney viscosity at ML 1 + 4 100°C	45	46	46.4	48.5	46	46.5	46	48
Scorch time (min)	9.0	8.3	10.3	10.3	9.3	11.0	12.0	14.3
<b>HAF Mix</b>								
Tensile strength (kg/cm <sup>2</sup> )								
unaged	279	273	270	270	268	269	268	275
aged (%)	94	94	95	97	93	94	91	91
Elongation (%)								
unaged	540	520	560	560	540	530	570	550
aged (%)	89	87	84	80	80	85	81	82
Modulus 300 (kg/cm <sup>2</sup> )								
unaged	125	127	115	116	119	122	110	117
aged (%)	134	138	150	153	143	141	155	144
Resilience (%)	67.6	67.6	65.4	66.3	67.0	67.2	66.1	65.4
Hardness (IRHD)	60.4	60.2	61.5	61.3	60.0	59.7	62.0	61.1
Tear strength (kg)	35.3	37.9	36.3	37.8	36.7	38.3	33.6	33.6
Akron abrasion (cc loss/100 rev)	0.10	0.10	0.10	0.11	0.11	0.11	0.11	0.11
Flex cracking	140	120	120	110	70	130	110	130
Cut growth (kc), 4 - 8 mm	126	103	102	103	136	96	103	112
Mooney viscosity (ML 1 + 4 at 100°C)	64.5	64	62.5	65	60	63.5	59	62
Scorch time (min)	20	19	18.8	19.2	20.2	20	20	21.2
Heat build-up (Goodrich)								
°F after 25 min	222	223	225	227	233	224	226	224
60 min	227	226	230	233	238	228	230	229
180 min	263	267	280	286	267	276	274	278
Permanent set (%)	30.3	28.5	38.5	36.1	29.0	31.7	33.2	33.0

Details of the S/S coagulation are the same as in Table 3.

coagulation system is adopted, being only a quarter of that for the latter (Table 5).

The cost difference is less when ammoniated latex is used and is only marginal for latex

TABLE 5. COST OF DIFFERENT COAGULATION SYSTEMS<sup>a</sup>

Ammonia (%)	Type of process	Time of coagulation (h)	pH of coagulation	Acid coagulation cost (c/kg)	S/S Coagulation		
					Surfactant (%)	Salt (%)	Cost (c/kg)
Nil	5L	8	6.7	0.26	0.05	0.10	0.24
		20	6.7	0.26	0.01	0.10	0.06
		20	6.7	0.26	—	0.15	0.03
	CV	8	6.5	0.24	0.01	0.20	0.09
		20	6.5	0.24	—	0.15	0.03
0.1	5L	8	8.5	0.92	0.10	0.10	0.66 <sup>b</sup>
		20	9.0	0.92	0.07	0.10	0.33
	CV	8	7.0	0.89	0.10	0.10	0.86 <sup>b</sup>
		20	7.5	0.89	0.10	0.10	0.77 <sup>b</sup>

<sup>a</sup>The cost of acid, surfactant and salt was taken as \$1.00, \$3.30 and 22 cents per kilogram respectively.

<sup>b</sup>The cost is inclusive of that of acid used for partial neutralisation.

treated with HH. Further reduction in cost can be achieved for non-ammoniated latex if the surfactant is deleted and 0.15% salt alone used.

#### DISCUSSION

The development of an instant coagulating system using di-octylsodium sulphosuccinate and calcium chloride has offered possibilities of affecting improvements in conventional coagulation. It has been found that both ammoniated and unammoniated latices can be completely coagulated by this method in batch-wise conventional coagulating tanks. JOHN (1971) reported that instant coagulation is best effected at near neutrality (pH 7.0) being extremely slow above pH 8.0; for ammoniated latex therefore prior neutralising with acid was required. The present work, however, has shown that latex ammoniated to pH 8.5 and pH 9.0 can be completely coagulated by the S/S method in 8 and 20 h respectively. Partial neutralisation to pH 7.5 is nevertheless required for latex treated with HH. The method is effective throughout the d.r.c. range normally encountered.

The addition of salt (0.3%) in instant S/S coagulation increased the ash content

of the resulting rubber (JOHN, 1971), sometime even exceeding the permissible level for whole latex rubber under the SMR specifications (MORRIS, 1970). In the present investigation where a longer period of coagulation was employed, even 0.5% salt does not markedly increased the ash content. It would therefore be reasonable to assume that ash content increases only if gelling takes place instantly. When latex coagulates instantly at a near-neutral pH, it is possible that the salt becomes occluded in the coagulum, whereas when gelling is gradual the salt remains in the serum. It is evident from the present studies that latex can be coagulated with up to 0.5% salt without adversely affecting the ash content, provided the process is allowed to take place gradually and not instantly.

The cost of producing rubber with S/S coagulation compares favourably with that of acid coagulation. Unammoniated latex, with or without HH, can be coagulated in 20 h at a quarter of the cost for acid coagulation; the cost can be further reduced to one-eighth by omitting the surfactant but by using a slightly higher concentration of salt. Thus, by coagulating fresh or ammo-

niated latex in batch-wise conventional tanks using very low concentration of surfactant and salt, it is possible to produce a good quality rubber at a reduced cost.

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