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#### INTRODUCTION

Sulphamic acid is a monoamide of Sulphamic acid. It is produced commercially by reacting urea, sulphur trioxide and sulphuric acid. Its principle applications are in metal cleaning, scale removers, detergent manufacture, stabiliser for chlorine and washing of felts<sup>1</sup>. The salts of sulphamic acid are used in flame retardants, weed and brush killers, synthetic sweetners and for electroplating.

Sulphamic acid is a dry, non-volatile non-hygroscopic, odourless, while crystalline solid. Stability is the outstanding physical property of the acid. It can be used as a primary standard for titrimetry.

In aqueous solution it is highly ionised and strongly acidic. The pH of 1% solution is 1.18. Sulphamic acid is moderately soluble in water. 14.68 grams will dissolve in 100 grams water at 0°C and 47.08 grams at 80°C<sup>2</sup>. Sulphamic acid crystallises in the othorhombic system with a unit cell of eight molecules. Reactions<sup>3</sup> of sulphamic acid are as follows :

i) Sulphamic acid begins to decompose at 209°C. At 260°C decomposition produces sulphur dioxide, sulphur trioxide, nitrogen, water and other products.

ii) Dilute solution of Sulphamic acid are quite stable at room temperature. A higher temperature however hydrolysis takes place forming  $NH_4$  HSO<sub>4</sub> and  $(NH_4)_2SO_4$ .

$$MH_2SO_3H + H_2O - --> NH_4 HSO_4$$

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Sulphamic acid has been found to be useful as an additive in bleaching of pulp. It retards the degradation of pulp during bleaching and improves the strength properties.

Sulphamic Acid for

Improved Bleaching

At West Coast Paper Mills, for bleaching pulp of 22-24 Permanganate number, it was found that, due to slight change in bleaching conditions the pulp quality would significantly change, this became acute as the pulp production was increased from 60 tonnes per day to over 100 tonnes per day. It was necessary to find some way of obtaining pulps of uniformly high quality.

Sulphamic acid was tried in the Laboratory during bleaching of bamboo sulphate pulps. It was found that the optimum results were obtined when 0.1 - 0.15per cent Sulphamic acid on the basis of O.D.'pulp was used in the hypochlorite stages of the bleaching sequence C/E/HH/.

Mill scale trials were then carried out for long periods and an exhaustive study was made of the use of Sulphamic acid. An increase of 36% in viscosity value was obtained by using about 0.1% Sulphamic acid on the basis of BD. puln for the brightness level of 80 per cent. The strength properties of pulp shows significant improvement in tensile, burst and folding endurance. The runnability of the paper machine had improved and it was possible to increase the filler content in the paper.

$$\begin{array}{l} \mathrm{NH_2SO_3 \ NH_4 + H_2O \longrightarrow } \\ \mathrm{(NH_4)_2 \ SO_4} \end{array} \\$$

The rate of hydrolysis is regulated by concentration, temperature and pH. iii) Chlorine, bromine and chlorates oxidise sulphamic acid to Sulphuric acid and to Nitrogen.

iv) Nitrous acid reacts very rapidly and qualitatively with sulphamic acid yielding nitrogen gas.

$$\begin{array}{r} \mathrm{NH}_2 \mathrm{SO}_3 \mathrm{H} + \mathrm{HNO}_2 - - - > \\ \mathrm{H}_2 \mathrm{SO}_4 + \mathrm{H}_2 \mathrm{O} + \mathrm{N}_2 \end{array}$$

v) Hypochlorus acid at low temperature forms N-Chlorosulphamic acid.

$$\rm NHClso_3H + H_2O$$

vi) Pure nitrous oxide is obtained by the reaction of conc. HNO<sub>3</sub> with the sulphamic acid.

$$\begin{array}{rrr} \mathrm{NH}_2 \mathrm{SO}_3\mathrm{H} + \mathrm{HNO}_3 & & \longrightarrow \\ \mathrm{H}_2\mathrm{SO}_4 + \mathrm{N}_2\mathrm{O} + \mathrm{H}_2\mathrm{O} \end{array}$$

# Analysis of Sulphamic acid

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It is qualitatively determined by the reaction with sodium nitrite.

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 $Na NO_2 + NH_2SO_3H ---->$  $NaHSO_4 + N_2 + H_2O$ 

Blower and Arnold<sup>4</sup> utilised this reaction in rapid direct titration method using starch iodite external indicator.

By gravimetric method it can also be analysed where sulphamate oxidised to sulphate. A gasometric method<sup>5</sup> depending on measurement of Nitrogen evol ved can be performed.

#### Sulphamic acid in Chlorination Stage

Tobar<sup>6</sup> reports that the addition of 1-2% Sulphamic acid based on available chlorine during chlorination stage results in higher viscosity (about 25%) without affecting the brightness of pulp in bleaching of Kraft pulp by C/E/H/ sequence. He also observed an interesting phenomena that viscosity of bleached pulp increased with the increased temperature during chlorination stage when 2% sulphamic acid based on available chlorine was used.

However, Guide and Holmes7 opined that the addition of sulphamic acid during chlorination stage was not useful.

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Later the mill trials carried out by Robinson and Santayers<sup>8</sup> at Champions mill established that the use of 2-3 lbs. of sulphamic acid per ton of pulp during chlorination stage minimised the degradation of pulp due to high temp. and low pH and also permits the increased production. The addition of sulphamic acid did not increase the excess chlorine consumption. Strength of pulp were significantly improved at same brightness. They observed that the increased strengths were greater on Kraft hard wood than on Kraft pine. Recently the effect of Sulphamic acid on the chlorination of Southern pine Kraft pulp<sup>9</sup> was studied for different retention time, temperature and levels of application. It was found that the addition of 1.5 lbs, of sulphamic acid per ton of pulp improved the pulp viscosity with affecting the lignin removal.

# Sulphamic acid in hypochlorite stage

Guide and Holmes<sup>7</sup> carried out extensively both laboratory experiments as well as plant trials by incorporating the sulphamic acid during hypochlorite stage of C/E/H sequence. The use of about 2.25 to 2.5% sulphamic acid increased the tearing resistance by 26% and folding endurance by 13%. Later Tobar<sup>6</sup> reported that adding 2 to 6% sulphamic acid in hypochlorite stage in C/E/H sequence increased the pulp viscosities by about 25%.

They also observed that the higher temperature and lower pH during hypo did not adversely affect the pulp when sulphamic acid was used. Tobar<sup>10</sup> repeated again about the advantages of the Sulphamic acid used in Hypo stage. It eliminated more or less the degradation caused by low pH and it increased the pulp brightness without reducing the strength and also allowed a small saving in hypochlorite consumption.

The various investigations showed that small amount of 2 to 4% of Sulphamic acid addition in Hypo stage is very effective. It is interesting to note that the sulphamic acid concentration above 4% had no effect on the viscosity of the pulp. The increased concentration of sulphamic acid reduces the rate of bleaching. At concentration above 40% no bleaching occurs. This effect is explained<sup>7</sup> by the fact that chloro-sulphomates formed have such a low oxidation potential which is not engough to bleach the pulp successfully. It is reported<sup>7</sup> that sulphamic acid cannot stop the undesirable brightness reversion which accompanies low pH hypochlorite bleaching.

At bleach liquor pH (above 11.5) the sulphamic acid is not stable and decomposes into sulphuric acid and nitrogen. Hence the sulphamic acid is added at the mixer separately where hypochlorite is added.

# Side Effects

It is observed<sup>7</sup> that the use of sulphamic acid affects considerably the retension of dyestuffs. The basic dyestuffs are retained less effectively on pulps bleached in presence of sulphamic acid. Auromine was the worst, being retained 27% less on the sulphamic acid bleached pulp than on non-sulphamic acid bleached pulp. No change in retention of acid and direct dyestuffs as well as rosin retention was observed. The bleached pulps bleached in presence of sulphamic acid are more difficult to refine than pulps bleached without sulphamic acid.

#### **Reaction Mechanism**

It is interesting to know how the small amount of sulphamic acid added during hypochlorite stage reduces the degradation of pulp to such a large extent. The chlorine, hypochlorite, or hypochlorous acid iron reacts with ammonia or amino compounds to produce mono- and dichloromines. Similar analogus reactions take place between sulphamic acid and chlorine or hypochlorite to produce chloro sulphamates (i.e. chloromines). These compounds have been isolated and characterised in 1918 by Traub and Von Drathen<sup>11</sup>. These workers also recognised the bleaching power of the chloro-sulphamates. McCarthy<sup>12</sup> has described the stabilisation of chlorine with sulphamic acid eliminating its volatility but actually enhancing the bacterial properties. The reactions that are known to take place between hypochlorite and sulphamic acid can be written.

active. The chlorosulphates have lower oxidation potential than hypochlorous acid or hypochlorite. In other words chlorosulphamates are less aggressive to pulp than hypochlorite or hypochlorous acid. It is assumed that the chlorosulphamates might function as reservoirs for hypochlorite thereby preventing a high concentration of oxidising chemicals in the early stages of bleaching thereby reducing the oxidation of pulp. Whatever major degradation to pulp takes place in hypochlorite bleaching is due to the formation of hypochlorous acid. The so formed hypochlorous acid, during hypochlorite stage reacts with sulphamic acid to form chlorosulphamates which are less reactive. This is how the reaction of the sulphamic acid in the hypochlorite bleaching is explained.

Another reaction mechanism is proposed<sup>13</sup> for sulphamic acid effect in hypochlorite bleaching. The Sulphamic acid forms complexes with inorganic ions and compounds<sup>14</sup>. It is also known that heavy metals catalyse the de-polymerisation of cellulose by hypochlorite<sup>15</sup>. Therefore, it is suggested that<sup>13</sup> the role of sulphamic acid in hypochlorite bleaching is to reduce or to eliminate the catalytic effect of heavy metal contaminents in bleaching liquor. Such mechanism suggests that sulphamic acid would be more effective with calcium hypochlorite than sodium hypochlorite solution and calcium salts have been shown to exert a greater catalytic effect. This proposed mechanism is not appreciated much.

The West Coast Paper Mill is producing daily about 100 tonnes of B.D. bleached pulp against its rated capacity of 60 tonnes per day. This high production naturally forced to change the optimum conditions of getting a best quality bleached pulp. We had to raise the temperature from 40°C to around 50°C in hypochlorite stage. It was very difficult to control the pH during hypochlorite stage as there was no proper mixing

$$NH_2SO_3Na + NaOCI \longrightarrow NHCISO_3 Na + NaOH \dots$$
 (1)

In this reaction NaOH is produced.

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$$M_2SO_3H + HOCI \longrightarrow NHCI SO_3 H + H_2O \longrightarrow$$
  
-- Chloro Sulphamic acid is produced.

The reaction (2) takes place at lower pH where formation of HOCl occurs. The chlorine in N — chlorosulphamates (reaction 1 and 2) is still positive and and automatic pH control. We had to sacrifice the quality of bleached pulp to a certain extent due to schedule of high production. We had to face some troubles on paper machine on account of

(2)

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Mode of addition to pulp	Control test	Hypo + S. acid mix- ture 3 mt. waited & then added	Hypo added 3 mt. stirred then S. acid added	Hypo & S. acid simulta- neously added
Hypochlorite as				· · · · · · · · · · · · · · · · · · ·
chlorine added, %	5.5	5.5	5.5	5.5
Sulphamic acid				
on OD pulp %	· . <u>-</u>	0.25	0.25	0.25
Hypo consumed, %	5.47	5.07	5.09	5.1
NaOH consumed as				
buffer, %	1.8	1.48	1.48	1.37
Brightness, %	78.5	77.5	78.0	78.5
Viscosity CED (cP)	14.1	19.9	19.3	22.1
C.A. cP.	35.6	52.7	50.9	59.1
pH_of pulp after				
addition	9.1	9.5	9.2	9.75

TABLE I

slightly weaker pulp. In such a situation the use of small amount of sulphamic acid during hypo takes care of increased production of pulp, increased temperature and fluctuations in controlling the pH, so that final quality of bleached pulp is not at all affected.

#### Laboratory Experiments

(I) First few experiments were carried out to find out the optimum condition of adding the sulphamic acid during hypochlorite stage. Whether the sulphamic acid is to be first mixed with hypo and then added to pulp, or it should be added after the addition of hypo, or, both should be added simultaneously.

The chlorinated and caustic extracted pulp from the washer No. II was collected and the hypochlorite bleaching was carried out in polythene bottle at 40°C and 5% consistency. The sulphamic acid was added with different sequence of addition in all the experiments except the control. The results are recorded in Table I.

Best results are obtained when sulphamic acid solution and hypochlorite are added to pulp simultaneously or mix the two just before addition to pulp.

(II) Another set of experiments were carried out to find out in which bleaching stage or stages the addition of sulphamic acid gives best results in the C/EH/H sequence which we used to follow in 1965<sup>16</sup>. The amount of sulphamic acid added in each stage was 0.04% on pulp basis. The conditions used in each bleaching stages and results are given below in Table II.

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# TABLE II

Bleaching conditions and results of addition of sulphamic acid in different stages of C/EH/H sequence. Unbleached bamboo sulphate pulp,  $\rm KMnO_4$  No. 24.

**Constant conditions :** 

	The bleaching	Sulphamia
	pH	8.0
	Time, Hours	3
	Hypo added as Cl <sub>2</sub> , %	3.0
	Temperature. °C	$44 \pm 2$
	Consistency, %	5.0
c)		
	pH	8.0
	Hypo added as Cl <sub>2</sub> , %	3.0
	NaOH added, %	2.0
	Time, Hours	1
	Temperature, °C	$55 \pm 2$
	Consistency, %	5.0
b)	Caustic/hypo stage, (EH)	
	Cl <sub>2</sub> added, %	7.0
	Time, Hours	1
	Temperature, °C	27
	Consistency, %	3.0
a)	Chlorination stage (C)	

It is clear that the best results were obtained when sulfamic acid was added to final hypochlorite stage (Table II expt. 6.) It improved the viscosity by about 4 points (cP) compared to control test (Table II expt. 1). However, the use of sulphamic acid in C, EH, and C/EH when followed the use in the first stage also gave satisfactory results (Table II expt. Nos. 2, 5 & 7) but the consumption of sulphamic acid was higher. But no improvement in viscosity was observed when sulphamic acid was added in 'C' and 'EH' stages. It was concluded that there is no positive effect of sulphamic acid addition in chlorination stage in improving the viscosity of bleached pulp.

(III) Strength properties: In continuation of previous experiments, the strength properties of bleached pulp, bleached in presence of sulphamic acid were also found out and compared with the control test where no sulphamic acid was used. For this purpose, the caustic/ hypo extracted pulp was collected from Pulp Mill having brightness of 68% and bleached by hypochlorite with the addition of 0.04% sulphamic acid on pulp basis. The washed pulp was beaten in the Laboratory Valley Beater (T200m-45) and standard sheets were made on British Sheet Making Machine according to standard procedures (T205m-58). The sheets were conditioned and tested for strength properties. The bleached conditions and results are given in Table III.

#### TABLE III

Pulp evaluation	
Final hypochlorite stage (H)	
Constant condition	
Consistency, %	5.0
Temperature, °C	$44 \pm 2$
Time, Hours	<b>2</b>
Hypo added as Cl <sub>2</sub> , %	2.8
рН	9.8

Expt.	The bleaching stages where sulphamic acid	Sulphamic acid added on pulp	Bleach consumption	Bright- ness	Viscosity, C.A., cP.
No.	was added	%	%	%	
1.	Nil	Nil	10.53	80.0	16.6
2.	С. ЕН & Н	0.12	10.64	78.0	20.0
3.	С & ЕН	0.08	10.66	80.0	15.2
4.	С	0.04	10.92	79.0	17.0
5.	С&Н	0.08	10.43	78.5	22.5
6.	н	0.04	r0.35	<b>`79.0</b>	21.0
7.	ЕН & Н	0.08	10.56	77.0	21.5
8.	EH	0.04	10.69	78.0	17.0

		With sulphamic acid	Control
Hypochlorite consumed as Cl <sub>2</sub> , %	•••	2.45	2.50
Sulphamic acid added :			
(a) on available Cl <sub>2</sub> basis, %		1.43	Nil
(b) on pulp basis, $\sqrt[6]{6}$		0.04	Nil
Brightness, %	•••	84.5	80.0
Viscosity (CA), cP	•••	21.0	19.0
Strength properties:			
Basis wt., g/m <sup>2</sup>		60	60
Breaking length, km.	•••	3.95	3.92
Burst factor		36.7	32.9
Tear factor	•••	88.0	81.4
Folding endurance (DF)	• •••	124	68

From the above results it could be seen that with addition of 0.04% sulphamic acid on pulp basis equivalent to 1.43% on available chlorine in the hypochlorite stage, gives an increase in viscosity by 2 points (cP) and brightness of pulp treated with sulphamic acid was high (84.5\%) the strength properties are higher than in the control test.

(IV) Another experiment was also carried out to have a clear picture of the improvement in various strength of paper by the use of sulphamic acid.

The C/E pulp from the washer No. II was collected and bleached by calcium hypochlorite using 0.25% sulphamic acid on pulp basis. A control bleaching test was also carried out where no sulphamic acid was used.

The consistency and temperature during hypochlorite stage were 5% and 40  $\pm$  2°C respectively. The hypo bleaching results are recorded below in Table IV. Both the bleached pulps were beaten in the Laboratory Valley Beater and the freeness at different intervals of time was recorded (Table V).

TABLE V

Beating Experiment Control			% on OD cid added
Time min.	°SR	Time min.	°SR
0	15	0	15
<b>20</b>	<b>26</b>	27	<b>26</b>
28	35	48	35
38	45	51	44
46	55	67	54

Beating behaviour of the both pulps is seen that the sulphamic acid treated pulps are difficult to refine i.e. more energy is required to beat to particular freeness compared to the pulp where no sulphamic acid is used.

Then standard sheets were made on British Sheet making machine and the conditioned sheets were tested for strength characteritics. The strength properties with and without sulphamic acid was recorded in Table VIA.

The breaking lengths of the standard sheets in the case of pulp bleached in presence of sulphamic acid are conside-

TABLE IV

#### Bleaching results

		Control	0.25 sulphamic acid + hypo simultaneous ly added
Hypochlorite as Cl <sub>2</sub> added	<u></u>	6.0	6.0
Cl <sub>2</sub> consumed, %		5.97	5.7
NaOH as buffer consumed ,%	•••	1.95	1.37
Brightness, % Viscosity,	•••	82.5	81.5
(CED). cP.	•••	8.1	12.3
(CA), cP.	•••	17.9	30.3

rably higher than the pulp without the use of sulphamic acid. The improvement in folding endurance is also quite significant (Table VIA).

(V) Experiments also carried out in Laboratory to study whether we can eliminate addition of buffer (NaOH) in hypo stage so that we can reduce the bleaching time and cost. Caustic extrated pulp was taken and bleached in presence of sulphamic acid with buffer and without buffer and also Hypo + buffer and Hypo alone was tried. The bleaching results are recorded below in Table VIB.

Consistensy %		5	%
Temperature		45	°C
Chlorine added	•••	6	%
Bleaching time	•••	120	min.

The results indicate that sulphamic acid is quite effective in preventive the lowering of viscosity of pulp and this effect is maintained even when the pH during Hypo stage dropped to 7.1 which otherwise is the worst pH in Hypochlorite bleaching.

# PLANT TRIALS

The Laboratory results on the use of sulphamic acid in hypochlorite bleaching of the pulp showed clearly the significant improvement in strength of bleached pulp. The plant trials were carried out to confirm the results and implement on plant.

The bamboo unbleached pulp of 22-24 permanganate number (40 ml) is obtained by the sulphate process. The screened unbleached pulp is bleached in upward flow towers by C/E/HH/sequence to 78-80% brightness.

The elemental chlorine in the emulsion is mixed with unbleached pulp slurry in the chlorine pennsalt mixer. The pulp with chlorine enters the chlorination tower (C) where 45-60 minutes retention time is given. About 8-9% elemental chlorine (50-60% of the total chlorine demand) is added on the B.D. pulp. The pH of the chlorinated pulp slurry is around 1.0-1.2. The chlorinated pulp is washed on the rotary vacuum washer.

About 4-4.5% caustic soda based on the unbleached pulp (BD. wt. basis) is added to the repulper of the chlorine washer pulp. Then the pulp enters the caustic reaction tower (E) where the

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retention time is about 60 minutes and temperature about 60°C. The pulp is washed on the rotary vacuum washer.

About 6-7% calcium hypochlorite is added in the caustic extraction washer repulper and the pulp with hypochlorite enters the reaction tower  $(H_1)$  where the temperature and reaction time are 40°C and 60-90 minutes respectively. To the overflow of the III tower, 1-2% hypochlorite is added without washing the pulp and 60-90 min. reaction time is given in hypo tower  $(H_2)$ . The overflow of the pulp is washed on the washer and the pulp is stored in the high density storage tower where the temp. is about 35-40°C.

# Handling System of Sulphamic acid :

The sulphamic acid is obtained in white crystals from indigenous source (99% purity) in a polythene lined gunny bag. 140 gpl solution is made in a polythene lined drum from where it is added to the repulper of the II washer and also in the overflow of the IV tower. The use of sulphamic acid during chlorination was not carried out in the plant trial as the laboratory experiments did not show encouraging results.

Earlier, in this report it is mentioned that the temperature during hypochlorite was used to be maintained around 50°C to cope up with the high production. It was not possible to give sufficient retention time and pulp resulting in the lower brightness. The pu'p from the final washer was used to be taken to storage chest where small amount of hypo was added and desired brightness level was achieved. In other words the retention time in the towers was not enough. Hence a new high density (14% Cy) tower is built which serves as a storage as well as for giving extra retention time, if required.

#### Plant Trial 1

The temperature of the hypo stage was brought below 40°C. This has improved the viscosity of pulp and sulpham'c acid addition was also carried out to see the effect on the improvement of the overall quality of bleached up. The results are recorded in Table VIIA.

The Table VIIA indicates that even though the brightness is less by about 1.7%the viscosity is almost double and con-

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TABLE VIAStrength Properties of Pulps

°SR	Co	ontrol	Expt.		S	ulpham	ic acid	added
	26	35	45	55	26	35	44	55
Basis wi. g/m <sup>2</sup>	57.62	60 33	63.85	62.54	57.1	58.8	58.4	58.9
Breaking length, km	3.90	4 50	5.28	5.20	4.12	4.97	5.93	6.01
Strength, %	2.1	3.0	3.4	3.43	2.4	3.1	3.4	3.6
Tear factor	70.3	80.4	72.1	64.0	104.3	90.2	81.3	80.7
Burst factor	23.3	25.4	27.9	32.0	22.1	25.5	35.5	35.8
Folding endurance								
(double folds)	4	3	7	6	11	11	9	10

TABLE VIB					
	Only Hypo	Hypo + buffer	Hypo + Sulphamic acid	Hypo + acid +	Sulphamic Buffer
Final pH	6.2	8.3	7.1	8.1	
Brightness, %	78.5	78.0	78.0	77.5	
Viscosity, CED, cP	6.5	7.5	10.5	12.2	

# TABLE VIIA

Pulp Evaluation			
		Average March 1970 When temp. was 48±2°C	Sample collected on 23-4-1970 when temp. was low and sulphamic acid added
1. Brightness, %		79.7	78.0
2. Viscosity CA; cP	•••	22.5	41.8
3. Basis wt. g/m <sup>2</sup>		—	61.1
4. Breaking length, m.	•••	5037	6200
5. Strength %	•••	2.88	3.9
6. Tear factor		70.8	90.0
7. Burst factor	•••	32.6	41.4
8. Folding endurance (DF)	•••	8	64

TABLE VIIB

Date & shift	Pulp brightness %	Viscosity (CA)cP	Date & shift	Pulp brightness %	Viscosity (CA)cP
15-4-70	81.5	22.9	17-4-70	78.5	41.8
'A'	80.0	25.0	<b>'A'</b>	81.0	43.2
	78.5	26.8		81.0	44.1
	78.5	33.8		82.0	36.5
<b>'B'</b>	77.5	37.6	.В,	80.5	35.9
	78.5	41.5		81.0	30.0
	78.0	45.0		78.5	42.7
	79.0	28.5		78.0	45.6
16-4-70	74.0	40.0	18-4-70	78.5	38.2
<b>'</b> A'	75.5	42.1	<b>'A'</b>	77.5	38.5
	75.5	48.2		79.0	39.1
	78.0	42.7			
<b>'B'</b>	80.0	30.9		80.0	36.2
	78.0	30.0	19-4-70		
	80.5	27.4	<b>'A'</b>	77.0	40.3
	79.5	33.5		78.5	40.3
				77.5	45.0
				77.0	43.2
TOTAL	1252.5	556.5		1344.0	679.7
Average	Brightness :	78.48	Average	Viscosity :	32.27

siderable increase in other all pulp strength properties are prominent.

The sulphamic acid was added at the rate of one kg. per ton of pulp processed along with calcium hypochlorite, at the alkali washer repulper. The rate of pulp flow was 3,500 litres/minute. The viscosity determinations were carried out on the final washed pulp every two hours and the results obtained are recorded in Table VIIB.

Thus obtained results were compared with the previous month average viscosity and brightness results where no sulphamic acid was used.

The viscosity of the bleached pulp has increased considerably. The average viscosity of the pulp (Table VIIB) is 32.27 cP. When compared with the previous months average viscosity of the bleached pulp which was 22.2 cP, it can be seen that the viscosity of the pulp has increased by 45%. It may be mentioned here that the pulp strength has increased so much that the refiner loads had to be increased to maintain the required freeness.

The average brightness of the pulp is 78.48%. When compared with previous months average brightness of pulp 80.7%, it can be seen that the brightness is less by 2 points. But, the pulp is cleaner and brightness can be further increased without degrading the pulp seriously.

#### Plant Trial No. 2

With the idea to increase the brightness again another trial was carried out by slightly changing the addition of sulphamic acid. In this case the sulphamic acid was added in two places i.e. (1) 0.1% at the alkali washer repulper along with the hypochlorite and (2) 0.05% at the overflow of the 4th tower. The trials were carried out from 9-5-1970 'A' shift to 11-5-1970 'B' shift.

The sulphamic acid addition was stopped from 11-5-1970 to 13-5-1970.

Again the addition of sulphamic acid as mentioned above was started on 15-5-1970 to 16-5-1970.

The viscosity results obtained every two hours are plotted to see the trend of improvement.

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The average results of viscosity and brightness are given below :

Date	Viscosity, cP (CA)	Brightness	%
9-5-1970 to			_
11-5-1970	29.8	81.4	
12-5-1970 to			
14-5-1970	23.5	80.2	
15-5-1970 to			
16-5-1970	32.4	81.2	

The viscosity results of the bleached pulp, bleached in presence of sulphamic acid (0.15%) is higher by about 32 per cent than the pulp bleached without sulphamic acid. The brightness of pulp is more by one point in the case of pulps bleached in presence of Sulphamic acid than the pulps without the use of Sulphamic acid.

#### **Plant Trial No.3**

In order to confirm the effect of sulphamic acid on hypo stage again the addition of sulphamic acid was stopped on 6-7-1970 and after collecting some readings up to 11-7-1970 addition was again started.

The average results of viscosity and brightness are :

Date	Viscosity, (CA) cP	Brightness	%
6-7-70 to			
9-7-70	30.3	81.8	
12-7-70 to			
13-7-70	36.7	81.7	

# DISCUSSIONS

Sulphamic acid in aqueous solution is quite stable at room temperature. Therefore, solution of sulphamic acid can be easily stored in PVC lined vessels and metered as and when required. Sulphamic acid is not quite stable in hypochlorite solution at pH over 11.5. This may be critical when sodium hypochlorite is used, in the case of calcium hypochlorite such high pH may not be encountered. However. it is desirable thhat sulphamic acid is added to hypochlorite solution just before it 1s added to pulp. In this case maximum benefit is obtained (Table No. I).

Sulphamic acid has been found beneficial during bleaching in the chlorination and hypochlorite stages. For bamboo sulphate pulp the results have shown that it is very effective in hypochlorite stage. (Table No. 11-6). Here an addition of 0.04% of sulphamic acid on pulp was found to give high viscosily. Since the oxidation potential of sulphamic acid with hypochlorite is lower than that of the hypochlorite and hypochlorous acid a large amount of sulphamic acid not recommended. For bamboo sulphate pulp, 0.1 to 0.15 per cent of Sulphamic acid on pulp basis is quite sufficient in the hypochlorite stage to give optimum results.

The part played by pH in the hypochlorite stage is quite critical. A pH drop below 8 is very harmful for the pulp quality. With the use of sulphamic acid even at pH 7.1 the pulp quality was superior than where caustic soda was used as buffer to maintain pH over 8 (Table VIB).

The plant trials have confirmed the Laboratory findings. The improvement in pulp viscosity has been significant. As the temp. of the pulp slurry was lowered below  $40^{\circ}$ C the sulphamic acid gave still better results (Table VIIA). It has been noticed that as soon as sulphamic acid addition is stopped the pulp quality deteriorates.

#### CONCLUSIONS

- 1. The use of Sulphamic acid in the bleaching of Sulphate pulp has been found beneficial.
- 2. The best point of addition of Sulphamic acid is in the latter stage of bleaching after alkali extraction.
- 3. An addition of 0.1% of sulphamic on the basis of B.D. pulp is sufficient to give considerable improvement in the quality of pulp.
- 4. The best results were obtained when buffer (NaOH) was used in hypochlorite stage along with sulphamic acid. However, in the plant even in the absence of buffer and when pH of pulp was 7 the Sulphamic acid maintained the strength properties of pulp. When buffer was added in plant trial the time of bleaching increased to get the same brightness.
- 5. The improved quality of bleached pulp obtained by the sulphamic acid made it possible to increase the filler in the paper and also effici-

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ency of paper machine was better. Besides the above the paper properties were better as a result the rejection and rework was also less.

6. Hence sulphamic acid could be advantageously used in improving the quality and bringing down the cost.

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