

# A study on replacement of sodium sulphate by AQ-Large scale trial at Rohtas.

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## SUMMARY

Review of literature, results of laboratory scale investigations and large scale plant trial with AQ are discussed in the article. Laboratory scale investigations have shown that AQ is more effective in Soda pulping than in Sulphate pulping. Large scale plant trial with AQ under identical conditions have resulted in lowering permanganate number of unbleached and alkali filter pulps, and improvement in yield, brightness and physical strength properties of papers.

The Kraft pulping process has several well established advantages in providing good quality pulp, high chemical and energy efficiency etc. Moreover, it is capable of pulping any cellulosic raw material. But the deficiencies of the process in terms of inferior yield coupled with economic and environmental pressure provide strong incentive for further search of superior processes. The efforts have been aimed at alternative processes capable of eliminating the deficiencies of the Kraft process while retaining its advantages.

The importance of Soda process as a viable alternative was realized if however, the basic drawbacks of the process viz. low pulp yield, inferior quality, longer cooking time, high temperature and caustic charges could be overcome by the use of suitable additives.

## ANTHRAQUINONE AN AID TO PULPING

The suitability of a process incorporating very small amount of Anthraquinone-2-mono-sulphonate sodium salt (AMS) into the system was indicated in the year 1972 by Bach and Fiehn<sup>1</sup>. The additive was effective both in Kraft and soda processes and it resulted in improved yield, a reduction in rejects and accelerated delignification without any adverse effect on physical strength properties of the pulp produced. Holton in 1977 found that Anthraquinone alone instead of its derivative was extraordinarily

effective in pulping wood chips and was superior to (AMS)<sup>2</sup>.

Kraft liquor is already a moderately effective delignifying agent hence greater effectiveness of anthraquinone was expected in soda process and was established. Holton observed that the small amounts of Anthraquinone so dramatically accelerated the soda pulping process that it was thought that it could effectively compete with or surpass the sulphate process in some respect. Moreover its application does not require any special technique and equipments.

The concept of pulping by incorporating Anthraquinone into the system has drawn worldwide attention<sup>3,4,5,6,7,8,9,10,11</sup>.

It has eliminated or reduced the pollution caused by sulphur and its compound.

Investigations have also shown that the addition of Anthraquinone did not enhance the toxicity of untreated bleached kraft effluents and that no difference in treatability or effluent characteristics were observed between the two effluents<sup>12</sup>.

Research and investigations carried out in different parts of the world have corroborated the findings of Holton.

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Investigations carried out in different laboratories and mills of our country have revealed following facts about anthraquinone usage.

- Soda Anthraquinone process gives yield and pulp quality similar to Kraft level and better than soda level in case of pulping Eucalyptus and pine wood<sup>7</sup>.
- In case of pulping of Eucalyptus hybrid it was observed that for identical pulping conditions addition of anthraquinone resulted in reducing the Kappa number of pulp by 10-15. It was also observed that anthraquinone is more effective in smaller dosages. There was improvement in physical strength properties of the resulting pulp<sup>8</sup>.
- Work done on bamboo (*D. strictus*) at the Research Centre of West Coast Paper Mills reveals that the benefits in the terms of increase in yield, reduction in H-factor and lowering of sulphidity can be obtained (either singly or in combination) by the addition of as small as 0.05% AQ on the basis of raw material during kraft pulping<sup>1a</sup>.
- AQ is also effective on mixed tropical hardwoods. For 0.05% dosage, there was reduction in active alkali charge by 2.0 to 2.4% producing pulp of kappa number 32 approx. Simultaneously there was increase in yield by 1.5%. If active alkali charge is not reduced, there is 16% saving in H factor (cooking time reduced) for 31-32 kappa number. Simultaneously the gain in yield is by 1.2%. If active alkali charge or H factor are not reduced, for a constant kappa no. of 34 there is scope for reducing the sulphidity from 25 to 5% by using AQ. No adverse effect has been observed in the bleachability, beating characteristics and strength properties of the pulp obtained by AQ pulping. Black liquor properties are also not affected<sup>9</sup>.
- Anthraquinone is effective in reducing active alkali charge, H-factor and chlorine requirement in bleaching. At the same time it increases yield. But it was found economical only when active alkali charge was reduced for maintaining mill kappa number at the same level<sup>10</sup>. (Raw material—bamboo and mixed hardwoods).
- Plant trial with AQ at Central Pulp mills has indicated that use of anthraquinone during cooking helps to reduce reject percentage which in turn gives rise to higher pulp yield. It is possible to maintain a higher kappa number in pulp with easy bleaching characteristics. Cost

savings are possible as the sulphidity range as low as 12-15% can be maintained enabling replacement of costly salt cake by other less expensive sodium make up<sup>11</sup>.

#### LABORATORY SCALE INVESTIGATIONS AT CENTRAL RESEARCH LABORATORY, DALMIANAGAR.

Investigations were carried out in the laboratory to see the effect of anthraquinone on pulp yield, bleachability and strength properties of both Soda and Sulphate pulps of our normal commercial chips. Commercial chips consisting of 70% bamboo and 30% hardwoods were taken for laboratory trials. Soda and Sulphate cooks with and without anthraquinone were carried on chips from same lot and under identical cooking conditions, (Table I). The pulping was done in 15 litre capacity laboratory rotary digester. The cooked material was screened on a 7 mesh screen with water jet pressure. The material passing through the screen are termed screened pulp and that retained on the screen as the reject. Screened unbleached pulp yield and percentage rejects were evaluated. The unbleached pulp was bleached under conventional multistage bleaching system consisting of CEHH sequence. Total chlorine consumption and bleached pulp yield were also determined. The unbleached and bleached pulps were beaten in the laboratory valley beater to 45° S.R. freeness, standard sheets were made on the British Standard Sheet making machine and the sheets tested for various physical strength properties. The cooking data are presented in Table I and unbleached and bleached pulp characteristics in Table II.

Following inferences are drawn from the experimental results :

- There is increase in yield of screened unbleached pulp and bleached pulp in case of AQ pulping.
- Under identical conditions of cooking the reject percentage is reduced by the use of AQ. The reduction is 4.9 and 0.4 percent respectively in soda and sulphate process.
- Anthraquinone is more effective in delignification in soda pulping than sulphate pulping. For 0.05% charge of AQ the drop in permanganate is 4.7 units in soda pulping as compared to 1.8 units in sulphate pulping under similar cooking condition.
- The reduction in permanganate number by the use of AQ has resulted in lower consumption of bleaching chemicals. This reduction being 3.0 and 1.1 percent respectively in soda and sulphate process.

**TABLE—I COOKING CONDITIONS— Laboratory Experiments**

		<b>SODA COOK</b>		<b>SULPHATE COOK</b>	
		<b>Blank</b>	<b>With AQ</b>	<b>Blank</b>	<b>With AQ</b>
1.	Moisture in chips, %	10.2	10.2	10.2	10.2
2.	Alkali on B. D. chips (as NaOH) %	22.0	22.0	20.0	20.0
3.	Chips liquor ratio	1:3.2	1:3.2	1:3.2	1:3.2
4.	Sulphidity in white liquor %	nil	nil	21.6	21.6
5.	AQ addition on b.d. chips %	nil	0.05	nil	0.05
6.	Cooking conditions :				
	a) Temp. raising time, mts.	75	75	75	75
	b) Max. temp., °C	162	162	162	162
	c) Time at max temp. mts.	120	120	120	120
	d) Relieving time, mts.	30	30	30	30
	e) Total cycle, mts.	225	225	225	225
7.	Screened unbleached pulp yield %	36.0	40.0	42.0	43.3
8.	Rejects	9.5	4.6	2.4	2.0
9.	Permanganate number	27.0	22.3	19.6	17.8

**TABLE—II. LABORATORY SCALE INVESTIGATIONS  
UNBLEACHED AND BLEACHED PULP CHARACTERISTICS**

	<u>SODA COOK</u>		<u>SULPHATE COOK</u>	
	<u>Blank</u>	<u>with AQ</u>	<u>Blank</u>	<u>with AQ</u>
<b>Unbleached Pulp Characteristics :</b>				
1. Initial freeness, °SR	10	10	10	11
2. Physical strength Properties at 45 °SR				
a) Breaking length, m	4500	4750	5000	5100
b) Burst Factor	35	34	38	37
c) Tear Factor	76	77	78	77
d) Double Folds	200	180	218	247
3. Total chlorine consumption during multistage bleaching consisting of CEHH sequence %	14.6	11.6	10.1	9.0
4. Brightness of bleached pulp (Elrepho),%	71.0	72.2	74.0	74.6
5. Bleached pulp yield,	31.8	35.9	38.1	39.4
<b>Bleached pulp characteristics :</b>				
6. Initial Freeness, °SR	13	14	14	14
7. Physical strength properties at 45 °SR				
a) Breaking length, m	4700	4800	4900	5200
b) Burst Factor	36	36	38	39
c) Tear Factor	68	71	70	72
d) Double Folds	175	170	200	210

— The strength properties of pulp are more or less unaffected by the use of AQ.

— The soda-anthraquinone pulping results are nearer to normal sulphate pulping.

#### PLANT TRIAL WITH AQ :

On the basis of investigational results large scale trial in the mill, with a view to eliminate the use of sodium sulphate was undertaken. Accordingly, two tonnes of Anthraquinone was procured from M/s. Indian Dyestuff Industries. Nearly 4000 tonnes of bone dry chips were cooked during the trial. The anthraquinone corresponded to 0.05% on b. d. chips was charged. The furnish during the trial was 70% Bamboo and 30% hardwoods. Anthraquinone was added to the digester when half of the digester was loaded with the chips. Other operational conditions of cooking and bleaching were similar to those maintained during normal sulphate pulping (Table III). The use of sodium sulphate in the soda recovery plant was completely stopped during the trial period. The sulphidity dropped to 3.0% during the trial.

TABLE—III CHEMICAL CONSUMPTION, COOKING AND BLEACHING CONDITIONS Plant Trial.

	With AQ	Without AQ
	(per tone of b.d. chips)	
1. Total alkali in white liquor (Consumption) kg.	194.6	198
2. Chlorine kg.	18.4	19.2
3. Bleach liquor kg.	24.2	22.4
4. Caustic for neutralisation kg.	5.14	6.0
5. Make up alkali, (caustic soda) kg.	28.7	26.5
6. Paper production kg.	386	363
<b>Cooking conditions</b>		
Steaming Time, mts.	75	75
Time at max. temperature mts.	60	60
Max. temperature, °C	165	165

#### EVALUATION OF MILL PULP

The consumption of different chemicals on b. d. chips and pulp characteristics like permanganate number, copper no. and brightness are given in Table III and IV. Samples of both unbleached and bleached pulp were collected in the factory, round the clock. The composite samples were evaluated in the laboratory as per TAPPI Standards for various characteristics. The pulp evaluation results along with those of similar pulp produced during the period preceeding the AQ trial are incorporated in Table V. These comparative figures speak of the relevance of AQ addition. Summary of Paper and board characteristics produced with and without AQ pulp are given in Table VI.

TABLE—IV SUMMARY OF PULP CHARACTERISTICS

Characteristics	Without AQ	With AQ
<b>1. Unbleached pulp</b>		
a) Permanganate No. of unbleached pulp	17.4	15.7
b) Permanganate No. of alkali filter pulp	6.5	3.7
<b>2. Bleached pulp</b>		
a) Copper Number of bleached pulp	2.49	2.71
b) Brightness of bleached pulp, % (Elrepho)	70.6	73.1

#### DICUSSION AND RESULTS

The evaluation of pulp properties at 45°SR freeness reveals that addition of anthraquinone into the system results in :

- increasing the breaking length of both unbleached and bleached pulps by 35 and 22 percent respectively;
- increasing the burst factor of unbleached pulp by 7 percent;
- maintaing similar folding endurance in case of unbleached pulp;
- maintaining similar water retention value and beating time of both unbleached and bleached pulps.

Table III reveals that Anthraquinone addition has resulted in slightly lower alkali consumption, lower chlorine and caustic consumption for neutralisation though bleach liquor and make up alkali (caustic soda) consumption have increased. The benefit in yield is also observed as shown by increase in paper production from 363 kgs to 386 kgs per tonne of Bone dry chips. The use of anthraquinone has resulted in reducing the permanganate no. of unbleached pulp from 17.4 to 15.7 and that of alkali extracted pulp from 6.5 to 3.7 under identical conditions of chlorination and alkali extraction. The brightness of bleached pulp increased from 70.6 to 73.1% during anthraquinone trials.

TABLE—V. MILL PULP EVALUATION IN THE LABORATORY.

Characteristics	Unbleached pulp		Bleached pulp	
	Without AQ	With AQ	Without AQ	with AQ
1. Beating time mts. (to attain 45 °SR)	28.7	27.5	16.3	15.4
2. Fibre length index (at 45 °SR)	0.93	0.80	0.73	0.52
3. Water Retention value (at 45 °SR)	234	233	242	237
4. Strength properties at 45 °SR				
Breaking length, m	3930	5330	3670	4490
Burst Factor	35.3	37.8	29.3	28.2
Tear Factor	89	83	59	41.3
Double Folds	385	390	34	14

Table VI depicting summary of paper and board characteristics with and without anthraquinone shows improvement in brightness of all varieties of paper, improvement in burst factor of kraft papers and breaking length of poster and creamwove papers.

## CONCLUSIONS

Following conclusions have been drawn from the Lab. and mill trial with AQ :

1. Sodium sulphate can be replaced by Anthraquinone. The replacement of sodium sulphate with AQ leads to sulphurless pulping. Thus provide better environmental protection and ultimate solution to kraft odour problem.
2. Reduction in permanganate number of both unbleached and alkali filter pulp made with AQ.
3. Increase in paper production per ton of chips i.e. higher yield has been obtained with AQ.

Table—VI. SUMMARY OF PAPER AND BOARD CHARACTERISTICS WITH AND WITHOUT ANTHRAQUINONE.

Quality	Properties	Without AQ	with AQ
Duplex Board	Brightness, %	65.9	67.2
Plain Kraft	Burst Factor, (120 GSM)	18.5	19.1
Ribbed Kraft	Burst Factor, (33-35 GSM)	19.0	20.0
	Burst Factor, (39-60 GSM)	19.5	22.9
Poster Paper	Burst Factor, (23-29 GSM)	17.8	17.8
	Burst Factor, (36-38 GSM)	18.4	18.1
	Breaking length, m, MD	2575	2940
	CD	2010	2240
Creamwove	Brightness, %	68.6	69.0
	Breaking length, m, MD	2585	Above 3000
	CD	1805	Above 2200
	Brightness, %	68.4	70.2

4. Improvement in brightness of pulp and paper, higher burst factor of Kraft paper and improved bleached grade has been noticed.

5. Use of Anthraquinone does not require any special technique or handling equipment.

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