

# Formaldehyde: A Useful Bleaching Aid To Save On Bleach Chemical Cost

Sahoo Sarita, Tripathy D.K., Panigrahi J.C. & Harichandan A.K.

## ABSTRACT

Presently pulp and paper industries use additives in pulping, oxygen delignification and bleaching to enhance the reactions and reducing the processing costs. Simple member of the family of aldehydes (formaldehyde) and ketones (acetone) having a carbonyl group ( $-C=O$ ) are effective as additive to increase the efficiency of chlorine dioxide bleaching. The performance studies of these additives to bleach the pulp samples after ODL with our raw materials at two different furnishes (100% mixed hardwood and bamboo : MHW 20 : 80) have been carried out to a target brightness of  $89 \pm 1\%$  ISO. The addition and effect of brightness gain was utilized for the reduction of high cost bleaching chemicals like  $H_2O_2$  and  $ClO_2$  in subsequent bleaching stages.

The paper describes how formaldehyde can be useful in saving 10.4 kg/ton of 50%  $H_2O_2$  solution or 2.66 kg/ton of  $ClO_2$  in a bleaching sequence of CD-Ep-D. In the process, the resultant pulp quality was also found to be better with respect to brightness, viscosity and strength properties. A decrease in pitch content was observed alongwith a reduction of TSS in the combined effluent. An indication of cost aspect has also been covered in this paper. Acetone did not prove useful inspite of saving 12 kg/ton of  $H_2O_2$  (50% solution) and 4.18 kg/ton of  $ClO_2$  due to its high cost.

## Introduction

Formaldehyde, a simple organic solvent has a carbonyl group ( $-C=O$ ) which reacts with chlorites to release  $ClO_2$  (1-3). In practice, industrial process wastes much of the  $ClO_2$  by forming chlorite ( $ClO_2^-$ ) and chlorate ( $ClO_3^-$ ). Chlorite is normally formed in the chlorination stage by both ionic and free radical mechanisms. Chlorine dioxide ionises to chlorate ( $ClO_3^-$ ), chlorite ( $ClO_2^-$ ) and chloride ( $Cl^-$ ) where hypochlorous acid ( $HClO$ ) and chlorous acid ( $HClO_2$ ) are produced as reaction intermediates depending on pH (4, 5). Chlorite is also formed by reduction of  $ClO_2$  on reaction with phenolic groups in lignin to give phenoxy radicals by one electron oxidation.

Realising the high cost of  $ClO_2$  generation, it was aimed at converting the  $ClO_2^-$  or  $HClO_2$  formed in situ, to  $ClO_2$ , by use of two organic chemicals, i.e. formaldehyde and acetone. Both of these chemicals were oxidized to their respective carboxylic acid on reaction with  $HClO_2$  and liberated  $ClO_2$  as the byproduct. This conversion of inactive chlorite or chlorous acid to  $ClO_2$ , increases the bleaching efficiency thereby resulting in increased brightness of final bleached pulp. The

increased brightness and bleaching efficiency of  $ClO_2$  can translate into bleach chemical saving in further stages of bleaching.

A mill trial of aldehyde enhanced bleaching for 3 days in 2001 in a Canadian mill was also reported (3), wherein a saving of 10.8 kg. of  $ClO_2$  per ton of pulp at a target brightness of 89.2% was achieved. Therefore a study was undertaken to see the possibility of arresting the chlorite formation or conversely to regenerate  $ClO_2$  from chlorites so that bleaching efficiency and utilization of bleach chemicals are optimized.

Although acetone was found to be equally effective compared to formaldehyde but due to its high cost (Rs.72/kg.) and hazardous nature, the study was pursued with formaldehyde for a possible industrial application.

## Understanding the mechanism:

The study was carried out in a bleaching sequence of CD-Ep-D. In CD stage of pulp bleaching, both  $Cl_2$  and  $ClO_2$  are added to pulp simultaneously, first chlorine followed by  $ClO_2$ . Initially, molecular chlorine and hypochlorous acid ( $HOCl$ ) are in equilibrium with equal quantities present at pH 2 and  $25^\circ C$  (6). When  $ClO_2$  is added to pulp, it reacts with pulp transferring one electron to produce chlorite ion ( $ClO_2^-$ ). This chlorite is unreactive towards lignin. Under acidic pH conditions, it is

converted to  $HClO_2$  which oxidizes lignin, to form  $HClO$ . The formation of chlorite or chlorous acid represents a loss of oxidizing efficiency of  $ClO_2$  (7). A most plausible mechanism of various parallel reactions involving  $HOCl$ ,  $ClO_2$ ,  $ClO_2^-$ , aldehydes and the pulp in the CD stage is discussed below :

It is generally accepted that both ionic and free radical mechanisms are involved in chlorination of pulp. Ionic reactions are predominant with lignin and radical reactions with carbohydrates. Lignin acts as an effective radical scavenger by virtue of favourable reaction kinetics and protected carbohydrates from oxidation. This protective effect diminishes when lignin content decreases. Chlorine dioxide is therefore added to pulp to protect radical attack in the CD stage (6). Literature reveals that within few minutes after the chlorination stage, 32% of  $ClO_2$  is converted to active chlorite resulting in loss of oxidizing power (8). Addition of formaldehyde prevents the loss of oxidation potential of  $ClO_2$  by generating  $ClO_2$  from chlorites. The carbonyl group in formaldehyde converts the chlorous acid ( $HClO_2$ ) and chlorite ( $ClO_2^-$ ), formed in situ to give back the  $ClO_2$  (9). In CD stage, where chlorination is carried out at or below pH-2, one of the ionisation products of  $ClO_2$  i.e. chlorite  $ClO_2^-$  is maximum. Chlorite is also regenerated by the reduction of  $ClO_2$  on

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Pulp and Paper Research Institute, Jaykaypur 765 017, Dist. Rayagada (ORISSA).



With 1% acetone, iii. With 1% acetone + 40% less H<sub>2</sub>O<sub>2</sub>, iv. 1% acetone + 60% less ClO<sub>2</sub> and v. 1% acetone + 40% less H<sub>2</sub>O<sub>2</sub> + 50% less ClO<sub>2</sub>).

## RESULTS AND DISCUSSION

A higher brightness (90.7% ISO) was obtained when formaldehyde was mixed with ClO<sub>2</sub> and added to pulp than that by adding directly to pulp (88.9% ISO) and without formaldehyde i.e. blank (87.8% ISO). Hence, adding formaldehyde to ClO<sub>2</sub> was more effective than adding it directly to the pulp.

The dose of formaldehyde at 0.75% on O.D. pulp basis was found to be the optimum (with respect to brightness and viscosity) and detailed experiments on 400 gm. scale were carried out, at this dosing. Optimization of formaldehyde dose with respect to brightness has been indicated in Figure-1. Similarly, optimization of acetone by applying 0.75, 1.0 and 1.5% on O.D. pulp was done and the optimum dose of 1.0% on O.D. pulp was selected.

It was found that addition of formaldehyde in both CD and D stages reduced the brightness by 0.6% ISO i.e. 88.9% ISO compared to blank (89.5% ISO). After the formaldehyde dose was optimized in preliminary study, a detailed study (with 400 gm. of OD pulp) was carried out to optimize the dose of formaldehyde at the target brightness level of 89 ± 1% ISO.

### WITH FORMALDEHYDE :

The results of study on 400 gm. scale showed that with the optimized 0.75% dose of formaldehyde on O.D. pulp basis, the yield, brightness, viscosity and strength properties increased when added to both 100% HW and B + HW furnish. A decrease in the amount of fines fraction and DCM extractive content in the final bleached pulp was also observed (Table 1 and 2).

The brightness increased from 88.1% ISO to 89.4% ISO in 100% HW furnish and 88.5% ISO to 90.6% ISO in B + HW furnish. The viscosity increase was 13.4% with formaldehyde addition as compared to blank in HW pulp whereas it was 1.3% in case of B + HW pulp. The strength index went up from 1508 to 1663 in 100% HW furnish (Table 1) whereas in case of B + HW, the rise was 1716 to 1797 (Table 2). Yield increase was 0.2% in B + HW furnish but no increase was observed in HW furnish. The pitch or DCM extractive content was 42.4% less in

FIGURE 1: EFFECT OF FORMALDEHYDE DOSE ON FINAL BRIGHTNESS

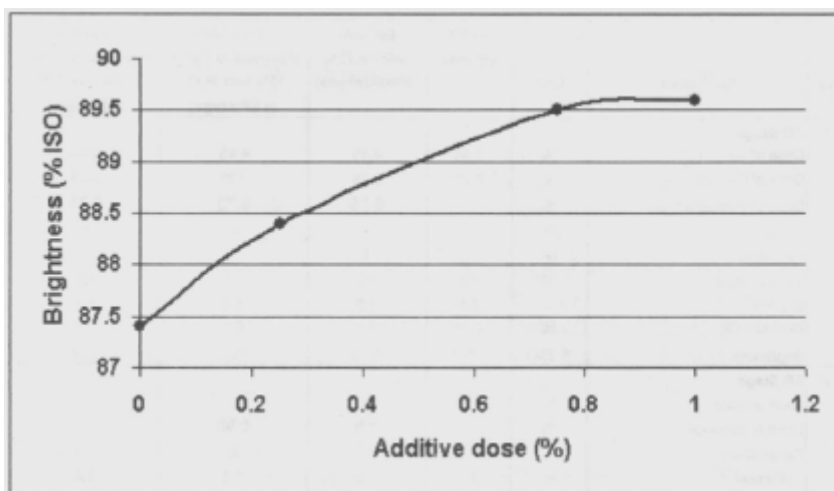


TABLE - 1: STUDY WITH FORMALDEHYDE (100% HW FURNISH)

PO Pulp unbleached kappa no. : 14.1

Sl. No.	Particulars	Unit	Set-I (Blank)	Set-II (With 0.75% formaldehyde)	Set-III (Formald. 0.75% + 35% less H <sub>2</sub> O <sub>2</sub> in EP stage)	Set-IV (Formald. 0.75% + 30% less ClO <sub>2</sub> in D stage)
1	<b>CD Stage</b>					
	Dose of Cl <sub>2</sub>	%	4.45	4.45	4.45	4.45
	Dose of ClO <sub>2</sub> as ClO <sub>2</sub>	%	0.25	0.25	0.25	0.25
	Dose of Formaldehyde	%	-	<b>0.75</b>	<b>0.75</b>	<b>0.75</b>
	Consistency	%	8	8	8	8
	Retention	hr.	1	1	1	1
	Temperature	°C	RT	RT	RT	RT
	End pH	-	1.8	2.1	1.5	1.9
	Residual Cl <sub>2</sub>	%	0.2	0.05	0.09	0.08
	Brightness	% ISO	52.3	54.3	54.8	54.1
2	<b>EP Stage</b>					
	Alkali applied	%	2	2	2	2
	Dose of peroxide	%	1.5	1.5	<b>0.98</b>	1.5
	Consistency	%	10	10	10	10
	Retention	hr.	1.5	1.5	1.5	1.5
	Temperature	°C	80	80	80	80
	End pH	-	10.7	10.4	10.7	10.3
	Brightness	% ISO	76.6	77.8	75.4	78.1
3	<b>D Stage</b>					
	ClO <sub>2</sub> applied as Cl <sub>2</sub>	%	2.2	2.2	2.2	<b>1.5</b>
	ClO <sub>2</sub> consumed as Cl <sub>2</sub>	%	1.9	1.96	1.75	1.36
	Consistency	%	10	10	10	10
	Retention	hr.	4	4	4	4
	Temperature	°C	70	70	70	70
	End pH	-	2.7	2.6	2.7	2.8
	Residual Cl <sub>2</sub>	%	0.3	0.24	0.45	0.14
	Brightness	% ISO	88.1	89.4	88.0	88.2
	Viscosity	cP	8.9	10.1	10.3	10.4
4	<b>Fibre classification :</b>					
	+ 16	%	0.1	0.1	0.1	0.3
	- 16, + 30	%	0.2	0.7	0.2	0.2
	- 30, + 50	%	34.7	36.3	33.2	32.7
	- 50, + 100	%	32.0	31.0	33.2	33.9
	- 100	%	33.0	31.9	33.3	32.9
5	Beating time to get 40°SR	min.	41	42	50	46
6	<b>Strength properties :</b>					
	Grammage	g/m <sup>2</sup>	61.9	62.1	61.9	59.7
	Thickness	micron	94.5	93.3	86.9	87.2
	Bulk	cc/g	1.53	1.5	1.4	1.46
	Burst factor	-	39.9	41.4	46.4	45.6
	Tear factor	-	65	76	71	67
	Breaking length	mtr.	5924	5990	6688	6723
	Double fold	no.	21	29	38	29
	Strength index		1508	1663	1733	1647
	7	DCM extractive (pitch content)	%	0.33	0.19	-
8	TSS of combined effluent (CD:EP:D :: 2:1:3) ratio	ppm	1318	480	398	354

**TABLE - 2: STUDY WITH FORMALDEHYDE (20% BAMBOO + 80% HW)**

PO Pulp unbleached kappa no. : 14.1

Sl. No.	Particulars	Unit	Set-I(A) (Blank)	Set-II(A) (With 0.75% formaldehyde)	Set-III(A) (Formald. 0.75% + 35% less H <sub>2</sub> O <sub>2</sub> in EP stage)	Set-IV(A) (Formald. 0.75% + 30% less ClO <sub>2</sub> in D stage)
1	<b>CD Stage</b>					
	Dose of Cl <sub>2</sub>	%	4.45	4.45	4.45	4.45
	Dose of ClO <sub>2</sub> as ClO <sub>2</sub>	%	0.25	0.25	0.25	0.25
	Dose of Formaldehyde	%	-	<b>0.75</b>	<b>0.75</b>	<b>0.75</b>
	Consistency	%	8	8	8	8
	Retention	hr.	1	1	1	1
	Temperature	°C	RT	RT	RT	RT
	End pH	-	2.0	1.9	1.5	1.5
	Residual Cl <sub>2</sub>	%	0.21	0.5	0.2	0.2
Brightness	% ISO	55.2	55.5	52.7	53.4	
2	<b>EP Stage</b>					
	Alkali applied	%	2	2	2	2
	Dose of peroxide	%	1.5	1.5	<b>0.98</b>	1.5
	Consistency	%	10	10	10	10
	Retention	hr.	1.5	1.5	1.5	1.5
	Temperature	°C	80	80	80	80
	End pH	-	10.7	10.6	10.9	10.6
	Brightness	% ISO	77.1	79.6	75.8	77.9
3	<b>D Stage</b>					
	ClO <sub>2</sub> applied as Cl <sub>2</sub>	%	2.2	2.2	2.2	<b>1.5</b>
	ClO <sub>2</sub> consumed as Cl <sub>2</sub>	%	1.7	1.8	1.9	1.4
	Consistency	%	10	10	10	10
	Retention	hr.	4	4	4	4
	Temperature	°C	70	70	70	70
	End pH	-	2.9	2.9	2.5	2.9
	Residual Cl <sub>2</sub>	%	0.5	0.4	0.30	0.13
	Brightness	% ISO	88.5	90.6	88.4	88.7
	Viscosity	cP	7.7	7.8	9.8	9.0
	Yield	%	94.6	94.8	94.7	94.9
	Shrinkage	%	5.4	5.2	5.3	5.1
4	Total Cl <sub>2</sub> applied as Cl <sub>2</sub>	%	7.3	7.3	7.3	6.6
	Total Cl <sub>2</sub> consumed as Cl <sub>2</sub>	%	6.6	6.4	6.8	6.3
5	<b>Fibre classification :</b>					
	+ 16	%	0.2	1.1	0.2	1.2
	- 16, + 30	%	8.5	5.4	10.1	8.3
	- 30, + 50	%	35.8	28.9	32.3	30.3
	- 50, + 100	%	24.8	23.5	24.2	24.5
- 100	%	30.7	31.1	33.2	35.7	
6	Beating time to get 40 <sup>0</sup> SR	min.	40	39	37	41
7	<b>Strength properties :</b>					
	Grammage	g/m <sup>2</sup>	58.3	60.7	61.4	61.0
	Thickness	micron	88.4	91.8	89.1	86.2
	Bulk	cc/g	1.51	1.51	1.45	1.41
	Burst factor	-	48	49	52	51
	Tear factor	-	72	73	75	75
	Breaking length	mtr.	6323	6326	7166	6336
	Double fold	no.	29	42	57	39
Strength index	-	1716	1797	1899	1826	
8	DCM extractive (pitch content)	%	0.45	0.25	-	-
9	TSS of combined effluent (CD:EP:D :: 2:1:3) ratio	ppm	1385	491	409	364

**TABLE - 3: EFFECT OF ADDITION OF ACETONE IN HW FURNISH**

Particulars	Brightness (% ISO)	Viscosity (cP)	Yield (%)	Strength Properties				
				BF	TF	BL (mtr.)	DF (no.)	Strength Index
Blank	88.1	8.9	93.0	39.9	65	5924	21	1508
Acetone (1%)	89.4	9.5	93.8	40.5	63	5965	33	1571
Acetone + 40% less H <sub>2</sub> O <sub>2</sub>	88.1	11.6	95.0	49.3	71	7029	63	1847
Acetone + 60% less ClO <sub>2</sub>	88.4	10.1	94.2	49.1	69	6873	40	1757
Acetone + 50% less ClO <sub>2</sub> + 40% less H <sub>2</sub> O <sub>2</sub>	88.4	12.3	95.8	53.4	68	7605	74	1893

formaldehyde added pulp as compared to blank in 100% HW furnish and 44.4% in B + HW furnish. This may be due to removal of HexA from pulp in

acidic conditions by the degradation of HexA in presence of HCHO thereby improving the final brightness (2).

At a similar brightness level by the addition of formaldehyde to pulp with reduced H<sub>2</sub>O<sub>2</sub> or ClO<sub>2</sub> doses in the Ep and D stages, the viscosity was further improved to 15.7%, 16.8% (for HW) and 27.3%, 16.9% (for B + HW) simultaneously increasing the strength properties. The breaking length values were also increased to 6688, 6723 from 5924 (blank for HW) and to 7166, 6336 from 6323 (blank for B + HW) at reduced doses of H<sub>2</sub>O<sub>2</sub> and ClO<sub>2</sub>. The TSS representing the fraction of fines decreased in the combined effluent in all the experiments with formaldehyde in both 100% HW and B + HW furnishes.

**WITH ACETONE :**

An increase in brightness was observed from 88.1 to 89.4% ISO and 87.9 to 89.8% ISO respectively for HW and B + HW furnishes (Table 3 and 4). The viscosity increase was only 6.7% for HW pulp where as a significant increase of 13.4% was obtained with B + HW furnish. The strength index went up from 1508 to 1571 in HW pulp and from 1716 to 1869 in B + HW pulp. The pitch or DCM extractive content of acetone added pulps of both the furnishes was 0.16% against the blank (0.31% for HW and 0.33% for B + HW). The TSS in the combined effluent decreased to 534, 506, 594 and 586 ppm respectively in acetone added pulps compared to blank (1318 ppm). Also, at similar brightness level, the viscosity and strength properties of final pulp were increased very much at reduced dose of H<sub>2</sub>O<sub>2</sub>, ClO<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> + ClO<sub>2</sub> in Ep and D stages.

Viscosity increase against blank (%):

Particulars	HW	B+HW
Reduced H <sub>2</sub> O <sub>2</sub>	30.3	13.4
Reduced ClO <sub>2</sub>	13.5	19.5
Reduced H <sub>2</sub> O <sub>2</sub> + ClO <sub>2</sub>	30.2	28.0

The strength index increased from 1508 (blank for HW) to 1847, 1757, 1893 and from 1716 (blank for B + HW) to 1919, 1822, 1844 respectively at reduced dose of H<sub>2</sub>O<sub>2</sub>, ClO<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> + ClO<sub>2</sub> respectively.

**COST IMPACT :**

Efforts were made to compare the differences in the cost of bleaching by use of formaldehyde for each set of experiments due to saving H<sub>2</sub>O<sub>2</sub> and also ClO<sub>2</sub>. The values are given in Table 5 and 6.



**TABLE – 4: EFFECT OF ADDITION OF ACETONE IN B+HW  
FURNISH**

Particulars	Brightness (% ISO)	Viscosity (cP)	Yield (%)	Strength Properties				
				BF	TF	BL (mtr.)	DF (no.)	Strength Index
Blank	87.9	8.2	92.4	48	72	6323	29	1716
Acetone (1%)	89.8	9.3	93.7	49	72	6568	71	1869
Acetone + 40% less H <sub>2</sub> O <sub>2</sub>	88.4	9.8	93.9	47	83	6789	65	1919
Acetone + 50% less ClO <sub>2</sub>	88.5	9.1	93.8	50	76	6744	39	1822
Acetone + 50% less ClO <sub>2</sub> + 40% less H <sub>2</sub> O <sub>2</sub>	88.3	10.5	93.4	49	83	6563	37	1854

Addition of 7.5 kg/ton of 40% formaldehyde solution at CD stage, it is possible to save about 2.66 kg/ton of ClO<sub>2</sub> or 10.4 kg/ton of H<sub>2</sub>O<sub>2</sub> (50% solution) at Ep stage. The cost benefit analysis shows that with formaldehyde, a net savings to the tune of Rs. 63.80/ton of B.D. pulp can be made by reducing the H<sub>2</sub>O<sub>2</sub> dose. While the net savings in cost is Rs. 21.20 / per ton of B.D. pulp was obtained in saving ClO<sub>2</sub>.

The cost of acetone at a dose of 10 kg/ton of B.D. pulp is comparatively high (approximately Rs. 720/-) compared to the costs towards combined savings of 12 kg/ton of H<sub>2</sub>O<sub>2</sub> (50% solution) and 4.18 kg/ton of ClO<sub>2</sub> (Rs. 264 + 292.60 = Rs. 556.60/- / ton).

### CONCLUSION

Formaldehyde can be utilized as a possible bleaching aid to achieve optimum efficiency of ClO<sub>2</sub>. The resultant pulp by the use of formaldehyde or acetone is better with respect to brightness and other critical properties. The viscosity and strength

properties further enhanced at reduced oxidizing chemical doses at similar level of brightness to control. There is no yield loss and the overall cost for formaldehyde addition works out to be lower by Rs. 63.80/T of B.D. pulp on H<sub>2</sub>O<sub>2</sub> or Rs. 21.20/T of B.D. pulp in ClO<sub>2</sub>. Industrial application is possible with proper storage and dosing mechanism on which the institute is working upon.

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**TABLE – 5: COST BENEFIT DUE TO H<sub>2</sub>O<sub>2</sub> SAVING BY USE OF FORMALDEHYDE**

Sl. No.	Particulars	Unit	Usage	Value (Rs.)
1.	Rate of formaldehyde (40%)	Rs./kg	22.0	-
2.	Dosage	kg./T	7.5	165.00
3.	Rate of H <sub>2</sub> O <sub>2</sub> (50%)	Rs./kg	22.0	-
4.	Reduction in H <sub>2</sub> O <sub>2</sub>	kg./T	10.4	228.80
5.	Net saving (4-2)	Rs./T	-	63.80

**TABLE – 6: COST BENEFIT DUE TO ClO<sub>2</sub> SAVING BY USE OF FORMALDEHYDE**

Sl. No.	Particulars	Unit	Usage	Value (Rs.)
1.	Rate of formaldehyde (40%)	Rs./kg	22.0	-
2.	Dosage	kg./T	7.5	165.00
2.	Cost of ClO <sub>2</sub>	Rs./kg	70.0	-
3.	Reduction in ClO <sub>2</sub>	kg./T	2.66	186.20
4.	Net saving (4-2)	Rs./T	-	21.20