Computation of TOCl and AOX from Cl_2 Consumption

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ABSTRACT:– Eight different empirical equations, available in the literature have been examined for computation of Total organic chloride (TOCl) and Adsorbable organic halides (AOX). TOCl and AOX values calculated using some of the equations for hard woods namely eucalyptus, subabul, casuarina, acacia, enterolobium cyclocarpum and gmelina arborea and bamboo as well as bamboo-hard wood are presented. The results obtained are discussed in terms of raw material, pulping and bleaching sequences adopted in the experiments.

INTRODUCTION

Among the new environmental regulations in the last ten years, chloro-organic compounds, expressed as TOCl (Total organic chloride) and AOX (Adsorbable organic halides), are in the focus worldover including in India. While in some advanced countries like USA and Canada the toxicity limit of TOCl/AOX is still being debated (1-3), countries like Sweden, Germany and Finland (4-8), have fixed the limit to as low as ~0.1 kg/ton. In U.S.A. (9) the proposed AOX limit for existing non-TCF bleached kraft mill is 0.156 kg/metric ton pulp. TOCl is fixed to 2 kg/ton in India (10) required for analysis and detection, is firstly difficult to be achieved. Secondly, toxic compound, "DIOXIN" (PCDD and PCDF) in TOCl, may be contained in the 0.1 kg/ton. The matter is therefore quite complex.

It may be imperative to be watchful at present juncture and have uptodate information on TOCl/AOX issue.

The basic information on AOX including nomenclature, source, analysis methods, effluent treatment methods for reduction of AOX, its environmental effect and concern in Indian context were given in an earlier publication (1).

Empirical equation collected from literature have been examined, used here for calculation of TOCl/AOX and the results are discussed in the Indian context.

RESULTS AND DISCUSSION

Equations for Calculation of TOCl/AOX:

8 different equations reported in literature (1, 11-17) have been examined here (Annexure - I).

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Eqns. 6-8 are regression equations involving kappa no.

(Annexure - I)

Equations for calculation of TOCl/AOX (kg/ton)

a) Eqn. 1 (Ref.11)

$TOCl = k\ [A\ Cl_2 + (A\ ClO^-/2) + (A\ ClO_2/5)]$

b) Eqn. 2 (Ref.12)

$AOX = 0.1\ [A\ Cl_2 + 0.6 \times A\ ClO^- + 0.2 \times A\ ClO_2]$

c) Eqn. 3 (Ref.1)

$AOX = 0.1\ [A\ Cl_2 + 0.526 \times A\ ClO_2]$

d) Eqn. 4 (Ref.13)

$AOX = 0.086 \times A\ Cl_2 + 0.12 \times A\ ClO_2/5$

e) Eqn. 5 (Ref.14)

$AOX = 0.089 \times A\ Cl_2 + 0.134 \times ClO_2/5$
$+ 0.023 \times A\ ClO_2/5$

g) Eqn. 6 (Ref.15)

$AOX = -2.21597 + 9.85 \times 10^{-3}\ (\%\ A\ ClO_2)$
$+ 2.08587\ (A\ Cl_2\ /\ Kappa\ no)$
$- 8.5 \times 10\text{-}5\ (\%\ A\ ClO_2)^2$
$- 0.01676\ (A\ Cl_2\ /\ Kappa\ no) \times (\%\ A\ ClO_2)$

h) Eqn. 7 (Ref.16)

$AOX = 1.04 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2]$
$- 0.156 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2]^2$
$+ 0.0132 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2]$
$\times$ Kappa no.

f) Eqn. 8 (Ref.16)

$AOX = 0.032\ [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2]$
$\times$ (Kappa no.) + 0.028

Active Chlorine

The parameters involved for calculation of TOCl/AOX are:

Active Cl_2 = A Cl_2
Active hypochlorite = A ClO^- and
Active ClO_2 = A ClO_2

Active chlorine is same as "available chlorine" (17) which can be determined by standard iodometric method (18) where the chemical reactions are:

Chlorine containing chemical + KI + $H^+ \rightarrow I_2 + Cl^-$
$I_2 + 2Na_2S_2O_3 = 2NaI + Na_2S_4O_6$

The available chlorine is determined by adding a measured amount (50 ml.) of bleach liquor to an acidified (10 ml. of 20% CH_3COOH) solution of 20% KI (10 ml.), which liberates an equivalent amount of iodine. The free iodine is determined by titration with standardized sodium thiosulfate solution (0.1 N), using starch as indicator.

$$\text{Available chlorine (gpl)} = \frac{V \times A \times 35.46}{5}$$

where V = volume of $Na_2S_2O_3$
A = normality of $Na_2S_2O_3$

Empirical equations:

The chemical analysis of TOCl is a tedious and expensive tool for evaluation of TOCl. The various empirical equations available in literature are discussed below.

Laboratory studies have shown a linear relationship to be existing among amount of Cl_2, hypochlorite and ClO_2 added and the amount of TOCl formed (11) as:

$TOCl = k\ [A\ Cl_2 + (A\ ClO^-/2) + (A\ ClO_2/5)]$ (1)

Where A Cl_2, A ClO^- and A ClO_2 are the active chlorine, hypochlorite and chlorine dioxide respectively and k is a constant for a given pulp. The authors have used different k values, namely:

k	= 0.07 to 0.08	for kraft pulp at laboratory scale.
	= 0.1	for kraft pulp (minimum value at mill scale)
	= 0.14	for kraft pulp (maximum value at mill scale)
	= 0.105	for conventional O_2 bleached pulp.
	= 0.074	for pulp pretreated with NO_2.

This empirical formula has been used (12) recently for calculation of AOX generated from the ECF bleaching sequence of O_2 delignified hard wood and bagasse pulps with k= 0.1 and multiple of A ClO^- as 0.6 instead of 0.5 in eqn-1.

$AOX = 0.1\ [A\ Cl_2 + 0.6 \times A\ ClO^- + 0.2 \times A\ ClO_2]$ (2)

Another relationship has been given (1) for estimation of AOX in the chlorination stage with same k value and multiple of $AClO_2$ as 0.526:

$AOX = 0.1\ [A\ Cl_2 + 0.526 \times A\ ClO_2]$ (3)

$AClO^-$ is not accounted as it was not used.

Lindstrom and Norden (13), from a series of bleaching experiments of delignified soft wood (pine and spruce mixture) pulp on the formation of AOX with Cl_2 and ClO_2 consumption, had shown that AOX formation is related to the use of Cl_2 and ClO_2 in the 'C' stage. The influence of ClO_2 application in final D or DED stages is insignificant. The authors have contradicted the eqn.-1 given by Germgard and suggested a regression equation of the type.

$$AOX = 0.086 \times A\ Cl_2 + 0.12 \times A\ ClO_2/5 \qquad (4)$$

The k value is not given separately here but it can be calculated as 0.086 with multiple of $AClO_2$ being 0.279. $AClO^-$ is not taken into account as it was not used.

Other workers (14) have suggested from their studies on the bleaching studies of soft wood with O_2 delignified pulp that the AOX formation from pulp bleaching reaction can be described through the following regression equation.

$$AOX = 0.089 \times A\ Cl_2 + 0.134 \times ClO_2/5 + 0.023 \times A\ ClO_2/5 \qquad (5)$$

Here also k value is not given but it can be calculated as 0.089 with multiples of $AClO_2$ in 1st bleaching stage as 0.301 and in 2nd stage as 0.052.

Another equation has been given by Basta et.al. (15) for calculation of AOX from the chlorine and chlorine dioxide charges according to the equation:

$$AOX = k_1\ [A\ Cl_2 + (A\ ClO_2\ /\ k_2)] \qquad (6A)$$

where $k_1 = 0.1$ and $k_2 = 5$

The significant way to lower AOX is to decrease the chlorine charge in the C stage. They have done a series of measurements of AOX after prebleaching an O_2 predelignified soft wood pulp (Kappa no. = 18) with different combinations of charge factors and degree of substitution of chlorine dioxide in the C stage and obtained a regression equation based on their results as follows:

$$AOX = -2.21597 - 9.85 \times 10^{-3}\ (\%\ A\ ClO_2) + 2.08587\ (A\ Cl_2\ /\ Kappa\ no) - 8.5 \times 10^{-5}\ (\%\ A\ ClO_2)^2 - 0.01676\ (A\ Cl_2\ /\ Kappa\ no) \times (\%\ A\ ClO_2) \qquad (6)$$

It is illustrated that pulp, prebleached with 20% A ClO_2 in the chlorination stage at % A Cl_2/ Kappa no of 2.5 gives 4.3 kg of AOX/ton. When % A Cl_2/ Kappa no. is constant an increase in ClO_2 from 20% to 100% will reduce AOX from 4.3 kg/ton to 0.95 kg/ton. When ClO_2 is kept constant at 20%, a drop in % A Cl_2 / Kappa no from 2.5 to 0.5 will reduce the AOX concentration from 4.8 to 0.8 kg/ton.

After comprehensive review of the literature, Tsai et.al. (16) have concluded that formation of AOX in the first two bleaching stages at atomic chlorine application rates of less than 3%, is best represented by equation:

$$\begin{aligned} AOX = {} & 1.04 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2] \\ & - 0.156 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2]^2 \\ & + 0.0132 \times [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2] \\ & \times \text{Kappa no.} \end{aligned} \qquad (7)$$

After multiple regression analysis on the experimental data against the same variables, the following equation has been suggested:

$$AOX = 0.032\ [\%\ A\ Cl_2 + 0.526 \times \%\ A\ ClO_2] \times (\text{Kappa no.}) + 0.028 \qquad (8)$$

Computation of TOCl/AOX :

The empirical equations can be used for computation of TOCl and AOX for any bleaching systems if A Cl_2, A ClO^- and A ClO_2 values can be known. These values can easily be calculated from Cl_2 consumption data.

In eqn. 1, k is reported to be 0.07 - 0.08 kg/ton for kraft pulps. it is indicated that k varies from mill to mill and from time to time. In one mill, k varied from 0.14 in one week to 0.10 in another week. In one pilot plant study, k was 0.105 (11). TOCl has been calculated with eqn-1 using (i) k = 0.075, (ii) k= 0.1 and (iii) k = 0.14 which will represent (i) for laboratory, (ii) minimum for mill and (iii) maximum for a mill.

TOCl and AOX have been calculated from the experimental results obtained previously (19-25) using CEH, CEHH and O C/D E D sequences with different hard wood and bamboo samples. From the bleaching conditions, the A Cl_2, A OCl^- and ClO_2 values have been calculated and are given in the Tables 2-4 along with TOCl and AOX values using the empirical equations.

Table-1

TOCl/AOX (Kg/Ton pulp) values with CEH bleaching of Eucalyptus (20)

Sample	Active Cl_2 (Kg/ton)	Active OCl^- (Kg/ton)	TOCl k=0.075	TOCl k=0.1	TOCl k=0.14	AOX
I	38.95	25.00	3.86	5.15	7.20	5.40
II	35.74	25.00	3.62	4.82	6.75	5.07
III	39.69	22.50	3.82	5.09	7.13	5.32
IV	36.51	15.38	3.32	4.42	6.19	4.57
V	38.30	18.83	3.58	4.77	6.68	4.96
VI	39.49	17.02	3.60	4.80	6.72	4.97
VII	35.80	20.94	3.47	4.63	6.48	4.84
VIII	34.35	20.74	3.35	4.47	6.26	4.68
IX	37.90	25.00	3.78	5.04	7.06	5.29
X	35.42	25.00	3.59	4.79	6.71	5.04
XI	35.12	25.00	3.57	4.76	6.67	5.01
XII	40.06	25.00	3.94	5.26	7.36	5.51
XIII	45.74	25.00	4.37	5.82	8.15	6.07
XIV	35.00	18.64	3.32	4.43	6.20	4.62
XV	40.00	22.84	3.86	5.14	7.20	5.37

Table-2

TOCl/AOX (Kg/Ton pulp) values with CEHH bleaching of other Hardwood (19-23)

Sample	Active Cl_2 (Kg/ton)	Active OCl^- (Kg/ton)	TOCl k=0.075	TOCl k=0.1	TOCl k=0.14	AOX
Enterolobium cyclocarpum	36.00	19.00	3.41	4.55	6.37	4.74
Gmelina arborea	82.00	19.00	6.88	9.18	12.85	9.37
Casuarina without bark	55.40	18.50	4.85	6.47	9.05	6.65
Casuarina with bark	61.00	21.50	5.38	7.18	10.05	7.39
Acacia without bark	48.00	17.00	4.24	5.65	7.91	5.82
Acacia with bark	51.00	17.50	4.48	5.98	8.37	6.15
Subabul I	50.00	28.00	4.80	6.40	8.96	6.68
II	39.30	28.60	4.02	5.36	7.50	5.65
III	42.00	19.90	3.90	5.20	7.27	5.39

Table-3

TOCl/AOX (Kg/Ton pulp) values with ClO_2 bleaching of delignified pulp (Bamboo : Hardwood) (21)

Sequence --		A	B	C
A Cl_2 (Kg/Ton)		35.6	35.6	35.6
A OCl^- (Kg/Ton)		--	--	2.5
A ClO_2 (Kg/Ton)		7.71	7.23	4.8
TOCl	Eqn 1	3.90	3.89	3.97
AOX	Eqn 2	3.71	3.70	3.81
	Eqn 3	3.96	3.94	--
	Eqn 4	3.24	3.24	--
	Eqn 5	3.26	3.26	--
	Eqn 6	5.03	5.03	--
	Eqn 7	2.20	2.05	--
	Eqn 8	1.31	1.31	--

Table-4

TOCl and brightness of bleached eucalyptus pulps

Sample	Brightness (% EI)	Sample	Brightness (% EI)	Sample	Brightness
I	82.3	VI	82.1	XI	79.6
II	81.7	VII	81.9	XII	80.3
III	80.3	VIII	82.3	XIII	79.9
IV	81.1	IX	79.2	XIV	80.7
V	80.1	X	78.9	XV	81.1

Table-5

Brightness of Hardwood pulps

Raw Material	Brightness (% EI)	Raw Material	Brightness (% EI)
Enterolobium	81.2	Acacia without bark	83.5
Gmelina arborea	82.4	Acacia with bark	80.4
Casuarina without bark	82.8	Subabul I	79.8
		II	80.8
		III	78.6
Casuarina with bark	80.3		

CEHH Bleached Pulp:

Table 1 contains 15 samples (20) of eucalyptus (tereticornis)collected from the same area (6 acres of land). It can be seen that the TOCl and AOX values vary from sample to sample. On changing k value, the TOCl value changes to almost double for the same sample. The average TOCl values and standard deviations for the 15 samples using different k values are as follows:

k value	Average TOCl (kg/t)	Average AOX (kg/t)	Standard deviation (kg/t)
0.075	3.67	--	± 0.279
0.1	4.89	--	± 0.371
0.14	6.72	--	± 0.536
0.1		5.11	± 0.393

The average TOCl value is 3.67 kg/t with the least k value and 6.72 kg/t with 'k' value of 0.14; the average AOX is 5.11 kg/t.

In case of casuarina, acacia and subabul (Table-2), these values are higher than in eucalyptus as the Cl_2 demand was higher in these hard wood samples (19 - 23). Subabul of 7 years old (III) somehow possesses lower TOCl value of 3.9 kg/t in stead of 4.8 kg/t in 3 years (I) old tree. However, the difference in TOCl value between 5 (II) and 7 years old subabul is little. Thus, from TOCl point of view also, 5 years old tree is quite suitable for pulping (20). Gmelina arborea (21) accounts for higher AOX/TOCl values compared to other hard wood. On the other hand, enterolobium cyclocarpum (22) has the minimum TOCl and AOX values; 3.41 kg/t of TOCl and 4.74 kg/t of AOX.

The TOCl value of acacia with bark (23), at k = 0.075 is 4.48 kg/t while it is 4.24 kg/t without bark; the corresponding AOX values are 6.15 kg/t and 5.82 kg/t. Thus the difference in AOX value with and without bark is not significant. In case of casuarina with bark, TOCl at k = 0.075 is 5.38 kg/t and without bark is 4.85 kg/t while the AOX value with bark is 7.39 kg/t and without bark, it is 6.65 kg/t. Here, the difference of TOCl and AOX between wood with bark and without bark is more significant than in acacia.

Catalyst treated pulp:

The TOCl values have been calculated (Fig.1) from the experimental results (24) of bamboo and bamboo-hard wood kraft pulps bleached with Molybdate catalysed CEpH sequence using eqn.-1 with k = 0.075. It can be seen here that the TOCl value for bamboo is 4.09 kg/t. The values from II to V (i.e. 4.23, 4.17, 4.02 & 4.13 kg/t) represent the CEpH bleaching of bamboo-Hard wood pulps. While II represents for the blank, experiments III - V represent for Mo catalyst with EDTA, Mo Catalyst without EDTA and uncatalysed with EDTA respectively.

FIG. 1: TOCl values with catalyst treated pulp

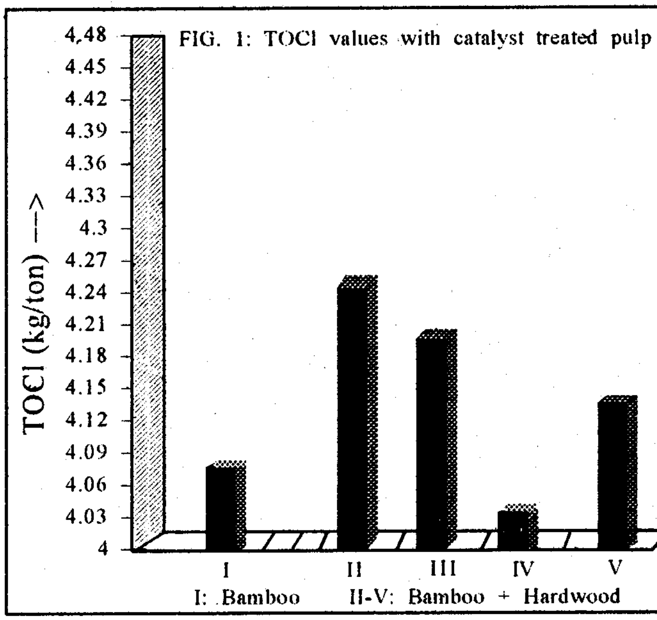
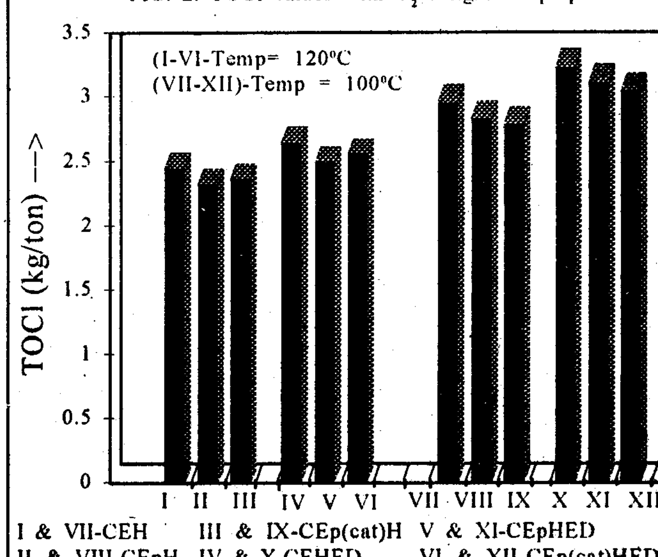


FIG. 2: TOCl values with O_2 delignified pulp



O_2- Delignified Pulp:

Experiments have been carried out (25) with bamboo-hard wood pulp different bleaching sequences such as CEH, CEpH, C Ep(cat) H, CEHED, CEpHED and C EP(cat) HED with oxygen delignification at 120° and 100°C in presence of Molybdate catalyst. The TOCl values of 2.09, 2.02, 2.07, 2.26, 2.18, 2.23, 3.06, 2.97, 2.91, 3.23, 3.13 and 3.08 kg/t (Fig. 2) shows that the TOCl values for experiments carried out at 120°C are lower than the experiments at 100°C for all bleaching sequences. The TOCl value for CEpH sequence at 120°C is found to be lowest (2.02 kg/t) while for CEHED at 100°C the value is high (3.23 kg/t). Because of low kappa no. after oxygen delignification, the Cl_2 demand has been quite low, resulting in low TOCl values.

ClO_2 Bleached pulp:

Experiments carried out with bamboo-hard wood pulp following in three bleaching sequences of oxygen delignified pulp using ClO_2 have been taken for calculation of TOCl and AOX from the 8 equations. It can be seen that according to eqn. 8, the least AOX value of <2 kg/t has been obtained. According to eqn. 7 also, low AOX value (2.05 kg/t) is possible; others have AOX/TOCl between 3 to 5 kg/t.

The AOX values reported in literature (26) with hard wood and bamboo-hard wood pulp following to various sequences show that AOX value is >4 kg/ton.

We have used constant "k" value in the equation for TOCl for all types of pulps though in fact, experimentally, it may be variable. The objective of this paper is computation of TOCl values from empirical equations from whatever bleaching data in the laboratory were available. The bleaching experiments had been carried out with different purpose. In Table-4 and Table-5 (19-23), the brightness values are given for different pulps which are quite variable. If the brightness would have been fixed to 75 or 80% El in all the cases, the TOCl values could have been different from the reported values; the k values could have been made to vary also accordingly. Further bleaching experiments will be carried out with varying Cl_2 and hypo doses, so that the minimum and maximum TOCl or "k" values can be obtained for different pulps.

AOX in pulp from a bamboo-hard wood based Indian mill was found to be 1.88 kg/ton which was calculated from the AOX value in the effluent. Because of the high volume of water in the effluent from Indian pulp and paper mills and use of imported soft wood pulp, such low figure should not be a surprise. In abroad, the volume of effluent (water) being low, comparison of AOX value should be made very carefully. It can be calculated as follows:

AOX analysed in effluent	= A ppm (mg/l)
Effluent volume of mill	= B M^3/day
Total production of pulp	= C ton/day.

$$\text{AOX in pulp} = \frac{A \times B}{1000 \times C} \; \frac{Kg}{ton} \qquad (9)$$

The effluent is to be sampled in glass bottle and to be adjusted to pH of 3 using concentrated HNO_3.

CONCLUSIONS

TOCl can be calculated on using the eqn.

$$TOCl = k[A\ Cl_2 + (A\ ClO^-/2) + (A\ ClO_2/5)]$$

where k= constant = 0.075, A Cl_2 = Active Cl_2, A ClO^- = Active hypochlorite and A ClO_2 = Active ClO_2.

The generation of TOCl depends upon raw material as well as pulping and bleaching conditions. The TOCl level with CEHH bleaching of hard woods and bamboo, is ~4 kg/t.

Pulp produced with CEHH bleaching, give higher TOCL values than pulp having oxygen delignification of O_2 +ClO_2 bleaching, With O_2-delignified pulp or O/C stage, TOCl value of < 2 kg/t is achievable. AOX in an Indian mill based on bamboo-hard wood was found to be 1.88 kg/ton of pulp.

ACKNOWLEDGEMENT

The authors express gratefulness to the Management of Pulp and Paper Research Institute, Jaykaypur for giving permission to publish this paper. The authors are also thankful to M/s. J.K. Corporation Ltd., Jaykaypur, Orissa for supplying of effluent sample and grateful to Dr. G. Lombardo of Centre technique du papier, Grenoble, France for AOX analysis.

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