Chelation with Magnesium Salt for enhancing Rayon Grade Pulp Quality

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Harihar Polyfibers, manufactures Rayon Grade Pulp by prehydrolysis sulphate process using eucalyptus hardwood. Rayon grade pulp is used in its fibre plants to manufacture viscose staple fibre. Higher iron content in the pulp was a problem hindering pulp brightness improvement & causing reversion. Mill conducted various studies for iron reduction by chelation. This paper presents process selection and its plant scale implementation whereby iron content in pulp reduced by 33% cost effectively with improved pulp quality.

INTRODUCTION

The Mill uses CEO/oHED bleaching sequence to get 90% ISO pulp brightness and controls iron content in final pulp by SO_2 treatment in last stage of bleaching. Still higher iron (Fe) content in the pulp to the extent of 18-20 ppm was a problem hindering pulp brightness improvement & causing reversion.

Mill investigated sources of iron such as wood, mill water, process chemicals and white liquor and found no cost effective solution to reduce iron in these process streams. Though mill used conventional EDTA chelation on plant scale and achieved 36% iron reduction, but with EDTA treated pulp the yellow colour of dyed fibre vanished at viscose staple fibre plant. The cost of EDTA treatment was also quite high i.e., Rs 70/ tonne of pulp. Hence EDTA treatment was discontinued.

Literature survey indicated that magnesium salts such as $MgSO_4$ are used for metal chelation during peroxide bleaching and oxygen delignification. Addition of $MgSO_4$ to an Eop stage or Ep stage prevents peroxide decomposition and protects pulp viscosity. Magnesium sulfate reacts with alkali and generates magnesium hydroxide ($Mg(OH)_2$), which inactivates transition metal ions present in the pulp by physically encapsulating them. With this, the transition metal does not get into contact & react with pulp resulting in improved pulp strength, increased delignification & increased pulp brightening. Metal- $MgSO_4$ chelate complex is water soluble & gets removed during post-treatment wash and hence no carryover problems. But there was no mill reference of $MgSO_4$ alone being used for iron reduction.

RESULTS AND DISCUSSION

Pilot Scale Study

To establish effectiveness of $MgSO_4$ in standalone condition, pilot scale trials were conducted using AR grade $MgSO_4$ in different locations in bleaching sequence. Extent of iron reduction and brightness / whiteness improvement with H_2O_2 + $MgSO_4$ and with $MgSO_4$ alone were studied.

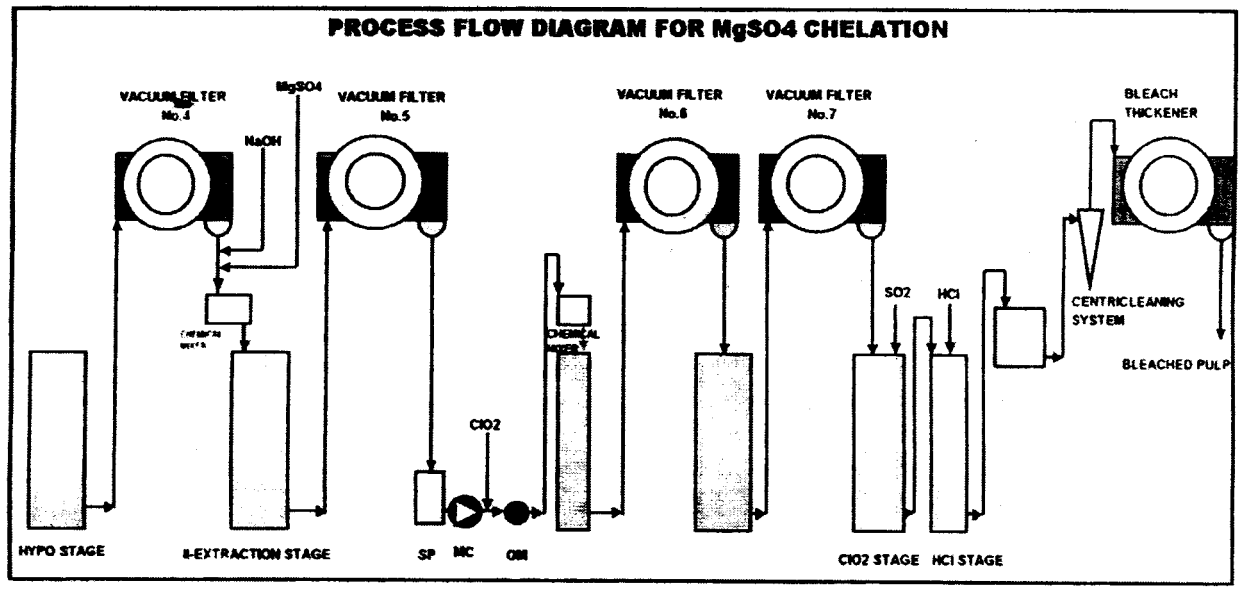
Table 1 : Process conditions for pilot scale trials

Stage	Consistency %	Temp °C	Retention minutes	pH
EO/o	11.0	70	110	11.5-12.0
E_2	11.0	60	60	10.5-11.0
After D	11.0	70	120	10.5-11.5

Legend : EO/o : Oxidative Alkali extraction, E_2 : Second Alkali extraction, D : ClO_2

Table 2 : Summary of Pilot Scale Trials with AR grade $MgSO_4$ & H_2O_2

Stage	H_2O_2 (100%) charge, kg/t	$MgSO_4$ charge, kg/t	Fe Reduction, %	Brightness Increase, units	Whiteness Increase, units	Cost Impact, Rs/tonne
EO/o	3.0	0.5	35.0	0.3	0.58	198
	Nil	0.5	21.1	0.2	0.40	30
E_2	3.0	0.5	62.7	1.63	2.53	198
	Nil	0.5	48.9	1.43	2.03	30
D	3.0	0.5	27.2	1.33	4.43	280
	Nil	0.5	22.2	1.10	1.53	30



PROCESS FLOW DIAGRAM FOR $MgSO_4$ CHELATION

Fig 1 : Process flow diagram of pulp chelation system with $MgSO_4$

Though iron reduction with $MgSO_4$ + H_2O_2 at second alkali extraction (E_2) stage was maximum, but was not cost effective. $MgSO_4$ treatment without peroxide showed good iron reduction efficiency and also cost effective. The same was studied with commercial grade $MgSO_4$ which showed similar reduction.

Plant Scale Trials

Plant scale trials were conducted with commercial grade $MgSO_4$. The $MgSO_4$ solution was dosed @ 0.5 kg /t at second alkali extraction stage along with caustic solution using a dosing pump. Pulp iron reduced from 18 ppm to 14 ppm i:e., 22% reduction but variation was on higherside. Investigation revealed that $MgSO_4$ solution concentration, dosing rate and inadequate mixing of $MgSO_4$ solution with pulp were causing higher variation. Unsettled $MgSO_4$ crystals at the bottom of dissolution tank were observed indicating incomplete dissolution of the salt. Pulp samples taken at different time intervals showed different iron levels indicating inadequate mixing of $MgSO_4$ solution with pulp. Plant scale trials were conducted and $MgSO_4$ solution dilution & dosing rate were optimized for better mixing. Same was regularized on plant scale from June 2003. The performance with and without $MgSO_4$ chelation is as under:

Conclusion

With addition of 0.5 kg/t $MgSO_4$ in second extraction stage, iron content in pulp could be reduced by 33%. This has helped in improving pulp and fibre quality. Additional cost of treatment is only Rs 3.0 per tonne of pulp. Thus mill has demonstrated that it is possible to reduce the iron content in rayon grade pulp cost

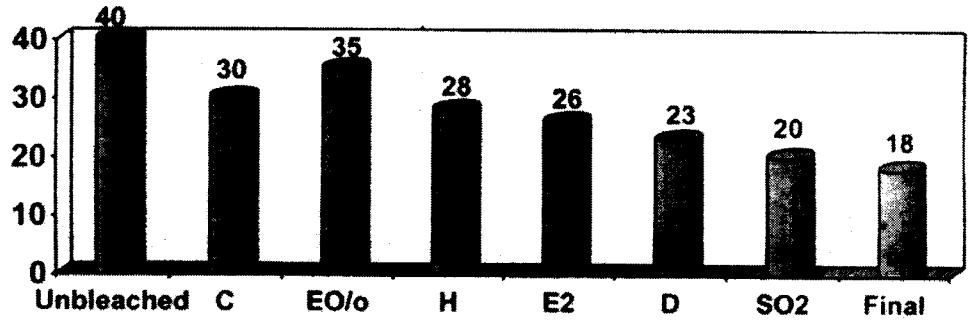


Fig. 2 Pulp Fe Without MgSO4 Chelation

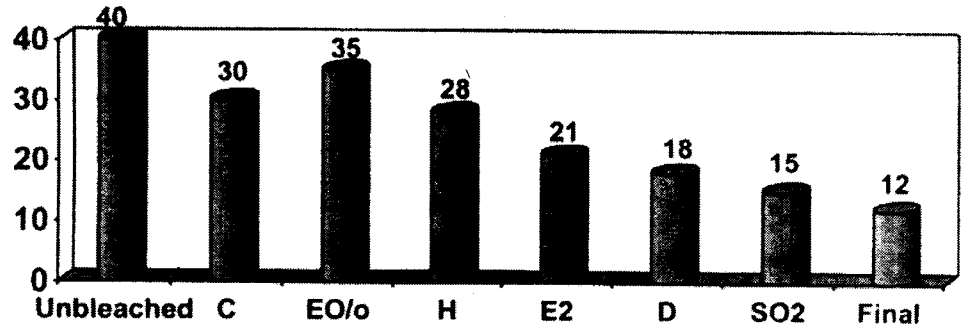


Fig. 3 Pulp Fe With MgSO4 Chelation

Table 3 : Improvement in pulp and fibre quality

Parameter	Unit	Without MgSO4 chelation	With MgSO4 chelation	Gain
Pulp Quality:				
Fe: Average	Ppm	18	12	6 (33% reduction)
Standard Devlation	-	1.34	0.99	0.35
Process Performance Index	Cpk	- 0.9	0.67	1.57
Brightness	ISO	90.0	90.8	0.8
Brightness reversion	%	2.3	1.6	0.7
Whiteness	Berger	77.0	78.2	1.2
Fibre Quality:				
Brightness	%ISO	87.3	87.9	0.6
Whiteness	Berger	70.8	71.8	1.0

effectively using magnesium salt thereby improving quality of viscose staple fibre

References

1. Z.Li, A.R.P. van Heiningen, Y.Ni, "Removal of Manganese from Pulp with $MgSO_4$ in a displacement system", Tappi Pulping Conf. 1999.

2. Carton W. Dence and Douglas W. Reeve, Editors, "Hydrogen Peroxide Bleaching", Pulp Bleaching: Principles and Practices, Tappi Press.