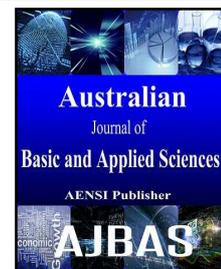




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Dissolved air flotation for recovering fibers from clear water of a paper machine.

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ABSTRACT

Background: Pulp and paper industries generate large volumes of wastewater. Approximately 40-30% of all fibers that enter the process ends in the whitewater and must be recovered and recycled to the paper machine, aiming at reaching maximum productivity and reduce wastes of raw material. The clear water studied in this research is the result of a primary treatment of white water wastewater through a filter disc equipment of a paper machine. **Objective:** The main objective of this paper is to investigate the use of dissolved air flotation (DAF), with the aid of cationic polymer, in order to remove fibers in clear water, generated in the production of two types of paper. **Results:** The use of cationic polymer allowed the formation of large, well-structured flakes, facilitating the flotation of total suspended solids (TSS) and reaching up to 99% of efficiency of removal. In the treatment of clear water from gloss paper, the best results of removal were 99%, 97%, 98% and 48.5% for TSS, apparent color, turbidity and chemical oxygen demand (COD) respectively. In the treatment of clear water from print paper reached removal efficiency values of 92.9%, 73%, 97% for TSS, apparent color, turbidity, respectively. Did not occur COD removal. The optimal configuration determined by laboratory tests was the dosage of 1 mg/L of cationic polymer and flow rate of 18 cm/min for both types of paper. **Conclusion:** The high efficiencies obtained in the research direct DAF with use of cationic polymer as an excellent treatment option for wastewater from pulp and paper industries, aiming to recover fibers.

INTRODUCTION

Recovery of water and fibers of the papermaking process, particularly of the paper machine system is configured in a cleaner production measure, of great economic importance and environmental. In recent decades, the pulp and paper industry followed the trend to adapt its production processes and operating with the main objective to reduce costs and waste, and improve their environmental performance (Pires *et al.*, 2013).

In the case of Kraft process, even for plants, which include the best production practices in terms of water consumption, yet consumption varies in the range from 40 to 55 m³/adt, which results in considerable volumes of sewage to treatment (BTAPP, 2011).

In the paper machine in the process of forming, pressing and drying the sheet, it is generated a so-called effluent "white water", mainly consists of high concentration of cellulosic fibers, mineral filler solids sedimented, suspended solids, starch, glue, thin fibers and organic matter, which increase the color and turbidity of the effluent (Sousa *et al.*, 2011).

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Approximately 40-30% of all fibers that enter the process ends in the whitewater and must be recovered and recycled to the paper machine, aiming at reaching maximum productivity and reduce wastes of raw material (Lacorte *et al.*, 2003).

In this context, it has been widely used in paper industries is the installation of dissolved air flotation (DAF) tanks as primary form of treatment. The dissolved air flotation stands out for its excellent removal of suspended and colloidal particles, with great potential in the treatment of white water and recovery of fibers, with typical values of removal of suspended solids from 70 to 98% (Miranda *et al.*, 2009). DAF is also able to reduce chemical oxygen demand(COD), color, turbidity and halogenated organic compounds present in wastewater (Ben *et al.*, 2004).

Since this is a quantitative research, the methodology included statistical analysis of the data that was essential to support the conclusions. We believe that the results presented in the manuscript are of great value to the scientific community of the area due approach to a real problem whose alternative technological treatment, the use of dissolved air flotation, consists of technically and economically viable solutions.

The main objective of this paper is to investigate the use of dissolved air flotation (DAF), with the aid of cationic polymer, in order to remove fibers in clear water, generated in the production of two types of paper.

MATERIALS AND METHODS

Characterization of the effluent:

This study was conducted in a medium-sized paper industry in the Midwest of the state of Paraná, Brazil, for business confidentiality reasons will not be nominated, which produces a total of 115.000 t/year of offset and kraft paper with wastewater flow estimated average of about 500 m³/day and concentration of suspended solids about 350-500 mg/L.

This is a case study, where the company already had a fiber recovery system, a filter disc, implanted in the flow chart of the paper machine, which, due to operational and technical problems, made the recovery efficiency around 60%, resulting in a loss of fibers and generation of excess sludge.

The volume of whitewater generated in the process, that passed through the filter disc, and was not reused in the process, resulted in a wastewater called clear water, which was used in this study.

Flotation tests:

The prior characterization of the clear water wastewater was carried out, which from now on will be called clear water, and the parameters analyzed were: pH, temperature, COD, turbidity, total suspended solids (TSS) and apparent color. All analyzes were performed according to Standard Methods for the Examination of Water and Wastewater 21 edition (APHA, 2005).

After the characterization, the clear water was heated to 37 °C to obtain the temperature under the same operating conditions during the production process. The pH was maintained in accordance with the natural characteristics of the collected clear water (7.8), to simplify a future deployment of DAF system in the industry, not being necessary to make the correction of pH. Further, it was subjected to treatment by coagulation/flocculation/flotation in a laboratory bench equipment, with batch operation, according to the methodology proposed by Di Bernardo and Centurione Filho (2003).

The operating conditions adopted were based according to values found in the literature (Costanzi and Daniel, 2002; Pioltine and Reali, 2011; Quartaroli *et al.*, 2014) and previously studied in a primary battery of tests performed in laboratory in order to investigate the feasibility of using a cationic polymer in the coagulation and flocculation process, since the same product is already used in industry as a retention aid in the wire mesh. All assays were performed in triplicate. The parameters set for all FAD tests are listed below in Table 1.

Table 1: Dissolved Air Flotation settings used for the tests.

Parameters	Configuration
Velocity gradient for rapid mix	500 s ⁻¹
Residence time rapid mix	60 s
Velocity gradient for flocculation	50 s ⁻¹
Residence time for flocculation	5 min
Recirculation	20% (v/v)
Saturation pressure	5 bar

It was tested a cationic polymer dose range of 0, 0.5, 1, 1.5, 2 and 2.5 mg/L to verify the performance of DAF. Along with the polymer dosage, it was tested two air flow rates (FR1 = 18 cm/min and FR2 = 9 cm/min), in order to optimize the process.

To evaluate the efficiency of removal of fibers, calcium carbonate, starch and other products used in paper composition, by using DAF, tests were made to determine the concentration of TSS, COD, turbidity and

apparent color of clear water. Within the specific study of recovery of fibers in the paper machine wastewater, was used the procedure for determining the concentration of TSS (Razali *et al.*, 2012).

The cationic polymer was tested with dilution of 5%. The product was given by the industry where the clear water was collected. Its physical and chemical characteristics are listed in Table 2.

Table 2: Physical and chemical characteristics of the cationic polymer.

Concentration	Poly Diallyl Dimethyl Ammonium Chloride 60% Coagulant Inorganic 30%
Physical State	Liquid
Color	Slightly yellowish
pH	3.0 – 6.0 a 10g/l (20 °C)
Density	1,0 – 1,2 g/cm ³ (20 °C)
Solubility	Soluble in water

Statistical analysis of data:

The study was done in a factorial arrangement 6 x 2 x 2 - completely randomized design, consisting of three factors. It was tested two different types of clear water, varying six doses of cationic polymer and two flow rates. Aiming to find correlation between dependent variables and the three factors studied it was decided that the response variables (dependent) chosen were: apparent color, turbidity, COD and TSS.

Statistical analysis and graphs were generated with version 7.0 software Statistica. The data were transformed (log) to fitgaussianity residue, verified by the Shapiro - Wilks test, homogeneity of variance, verified by Bartlett test. It was adopted a 5% significance level.

RESULTS AND DISCUSSION

Characterization of clear water:

Tables 3 and 4 show the physicochemical characterization of the clear water generated in the production of print and gloss paper, respectively.

Table 3:Physicochemical characterization of clear water from print paper.

Parameters	Print
Apparent color (HU)	1.104
Turbidity (NTU)	812
COD (mg/L)	446
TSS (mg/L)	545
Temperature (°C)	37

Table 4: Physicochemical characterization of clear water from gloss paper.

Parameter	Gloss
Apparent color (HU)	1.760
Turbidity (NTU)	544
COD (mg/L)	570
TSS (mg/L)	410
Temperature (°C)	37

Even after passing through the disc filter, the clear water still has a high concentration of fibers, which can be evidenced in the TSS values of 545 mg/L and 410 mg/L for print and gloss, respectively (Tables 3 and 4). Thus, there is great potential for recovery and reuse of fibers in case the compared values with those found in the tests carried out by Costanzi and Daniel (2002), where the clear water from a paper machine presented TSS concentrations of 112 mg/L. In this study two technologies to recover fibers were tested, the authors considered that DAF had a better performance compared to sedimentation.

The color of water sample is directly related to the intensity reduction degree that the light undergoes when crossing it, due to the presence of dissolved solids, especially material organic and inorganic in colloidal state. In this case the source of high color can result from effluents from pulp and paper industries, containing lignin, cellulose, starch and calcium carbonate (Di Bernardo *et al.*, 2011).

It was observed that the variation of polymer dose used in DAF tests does not significantly interfere in the pH value ranging between 7.5 and 7.9. Indeed appreciable when compared to other coagulants, which, by altering the pH of the wastewater (usually acidic range), those products demand high consumption for pH control and consequently produce large amount of sludge and raise the cost of treatment. It is also favorable for future reuse of the treated clear water, because the paper production use an alkaline process.

Removal efficiency of the studied parameters:

Table 5 and 6 show the percentage of the average removal efficiencies of the main variables studied in the treatment of clear water by DAF from print and gloss paper, respectively.

Table 5: Removal efficiency of parameters apparent color, turbidity, COD and TSS for DAF in the treatment of print paper clear water effluent.

Print Dosage	Apparent color (%)		Turbidity (%)		COD (%)		TSS (%)	
	FR 1	FR 2	FR 1	FR 2	FR 1	FR 2	FR 1	FR 2
0	57,0	60,0	89,2	90,1	-	-	70,3	70,6
0,5	70,0	70,0	97,2	97,0	-	-	76,8	78,6
1	73,0	73,0	97,2	97,0	-	-	90,7	92,9
1,5	68,0	68,0	97,9	97,9	-	-	94,3	94,6
2	73,0	73,0	98,0	98,4	-	-	85,6	87,8
2,5	73,0	75,0	97,1	97,1	-	-	76,1	84,7

Flow Rate 1 = 18 cm/min; Flow Rate 2 = 9 cm/min

Table 6: Removal efficiency of parameters apparent color, turbidity, COD and TSS for DAF in the treatment of gloss paper clear water effluent.

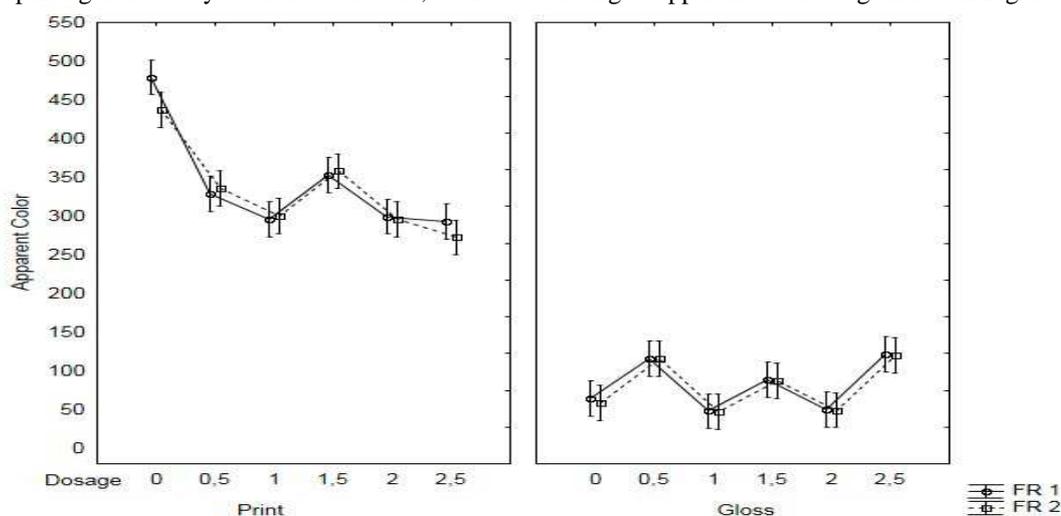
Gloss Dosage	Apparent color (%)		Turbidity (%)		COD (%)		TSS (%)	
	FR 1	FR 2	FR 1	FR 2	FR 1	FR 2	FR 1	FR 2
0	96,0	97,0	87,9	87,9	49,1	48,2	95,6	96,7
0,5	93,0	93,0	97,4	97,2	42,5	44,9	98,5	98,0
1	97,0	97,0	98,1	98,1	48,5	46,4	99,0	99,2
1,5	95,0	95,0	98,4	98,6	40,1	39,2	94,7	94,3
2	97,0	97,0	98,6	98,8	40,2	42,1	94,3	96,8
2,5	93,0	93,0	97,4	97,3	37,9	36,4	96,7	96,8

Flow Rate 1 = 18 cm/min; Flow Rate 2 = 9 cm/min

Analyzing the Tables 5 and 6 is evident that the float rate 1 and 2 showed similar performance in the removal of parameters. After treatment by DAF, the clear water from gloss paper (Table 6) obtained, in general, higher percentage removal of parameters than the results from the clear water from print paper (Table 5).

Apparent color:

Graphics generated by Statistica software, with data relating to apparent color are gathered in Figure 1.

**Fig. 1:** Interaction between: dosage x flow rate x paper type for the apparent color parameter.

According to Figure 1 we can say that there was no statistical difference between the flow rates tested. As regards the type of paper and the dosage of cationic polymer, assuming the flow rate FR 1 = 18 cm/min is most suitable as the volume of treated wastewater, over time, would be larger.

The best doses, statistically indicated, for both types of paper clear water were 1 and 2 mg/L (Figure 1). Considering saving polymer, it is preferable to choose the lowest dosage, since both have removal results of apparent color statistically equal.

As can be seen in Figure 1 the treatment of clear water from gloss paper had the best performance in removing apparent color (97%) at a dose of 1 mg/L to the FR 1 (Table 6) not statistically different from dosages 0.5, 2 and 2.5 mg/L. The clear water from print paper reached its best performance up to 75% removal at dosages of 1, 2 and 2.5 mg/L to the FR 1 (Table 5) which are statically equals.

These results were satisfactory and similar to those of Quartaroli *et al.* (2014) who used DAF with flow rate of 9 cm/min, aid of cationic polymer and polyaluminum chloride (PAC) in the coagulation process. Achieving 88% reduction of apparent color present in the wastewater from the secondary clarifier of an effluent treatment plant from a pulp and paper industry.

The clarification by DAF can be adopted when the raw water has a relatively high color in relation to turbidity; it facilitates the removal of small flakes, generally produced after the coagulation and flocculation (Di Bernardo *et al.*, 2011). As seen in Tables 3 and 4 where values of apparent color from clear water from print and gloss are respectively 1.104 and 1.760 HU compared with the turbidity of 812 NTU and 544 NTU.

Mathur *et al.* (2004) state that reduce effluent color before final disposal in water is an important goal. Flotation is shown as viable alternative for efficient clarification of clear water.

Turbidity:

Graphics generated by Statistica software, with data relating to turbidity are gathered in Figure 2.

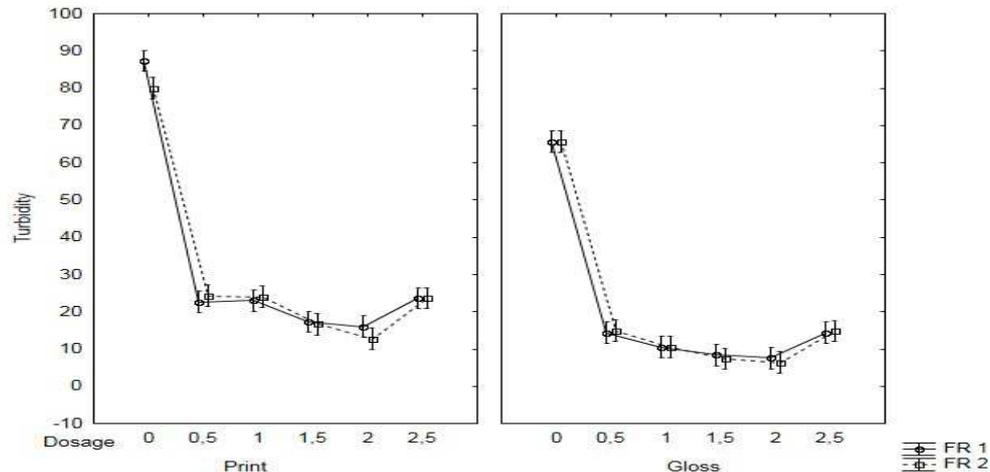


Fig. 2: Interaction between: dosage x flow rate x paper type for the turbidity parameter.

There was no statistical difference between the flow rates tested. For the clear water from gloss paper the turbidity removal reached 98% in the cationic polymer dosages of 1, 1.5 and 2.0 mg/L in the treatment of (Figure 2). Such dosages may be considered statistically similar to the dosages 0.5 and 2.5 mg/L with 97% removal efficiency (Table 6).

For the clear water from print paper the dosage of 2 mg/L had the best removal efficiency of 98% (Table 5), statistically similar to the dosages of 0.5, 1, 1.5, and 2.5 with 97% efficiency (Figure 2).

Costanzi and Daniel (2002) analyzed the turbidity removal behavior of the clear water effluent from a paper machine treated by DAF with the aid of ferric chloride. The authors obtained good turbidity removal efficiency for natural pH with the best value remaining 5.78 NTU, which resembles those obtained in the tests of this study utilizing cationic polymer, with remaining values of 6.2 NTU and 11 NTU to 2 mg/L dosage on clear water from gloss and print paper respectively.

Quartaroli *et al.* (2014) obtained a 90% reduction of turbidity, using DAF after treatment sludge waste activated in a pulp and paper industry. Assuming that the turbidity is related to the concentration of suspended solids present in the liquid, DAF shows an indirect way the potential for removing fibers, which consecutively are related with suspended solids.

COD:

Graphics generated by Statistica software, with data relating to COD are gathered in Figure 3.

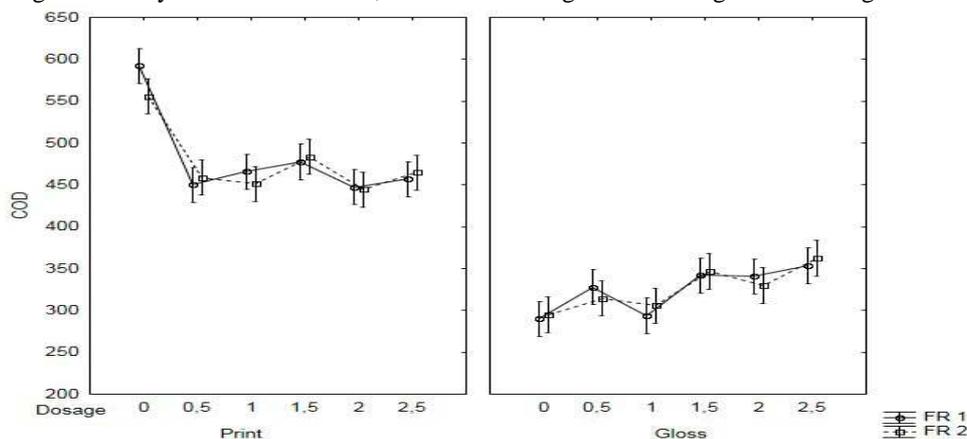


Fig. 3: Interaction between: dosage x flow rate x paper type for the COD parameter.

There was no statistical difference between flow rates (Figure 3), regardless of polymer dosage in both types of clear water.

There was no removing of COD parameter for clear water from print (Table 5). Dosages of 0.5, 1, 1.5, 2 and 2.5 mg/L were statistically similar (Figure 3). Possibly due to the fact that the COD is in soluble form in the effluent, confirmed by Belosinschi and Bobu (2007) reported that while the suspended solids are easily removed in primary treatment systems that exist on paper machines, much of the material dissolved colloidal remains in the effluent, in COD form.

Such results should not cause problems in relation to the remaining COD, as after passing the primary treatment of DAF, part of the clarified water would be reused in the system itself and another part would go to the effluent treatment plant of the company, which will be diluted and treated in the secondary treatment system, consisting of activated sludge.

Quartaroli *et al.* (2014), studying DAF treatment in secondary effluent from a pulp and paper industry, reported that residual values from tests with dosage greater than 1000 mg/L of cationic polymer increased the remaining amount of COD, thus proving that the polymer contributed to the increase in organic matter and that part the fibers may have been solubilized, generating mainly dissolved organic matter, represented by the increase of COD in the effluent. The COD is not always removed efficiently by being present mainly in soluble form (Sharma *et al.*, 2014).

The best performance in the removal of COD for clear water from gloss, reached removal of 49%, without the addition of cationic polymer, 48.5% for dosage of 1 mg/L and 44.9% for dosage of 0.5 mg/L (Table 6) which are all statistically similar. It is noted that without the use of cationic polymer the removal of COD was the same when using it, so the coagulation was not efficient for COD removal

The results are similar to those found by Woollenet *al.* (1995), which achieved 30 to 40% COD reduction by using a wastewater treatment system of the paper machine constituted of DAF followed by a sand filter and disc filter.

TSS:

Graphics generated by Statistica software, with data relating to TSS are gathered in Figure 4.

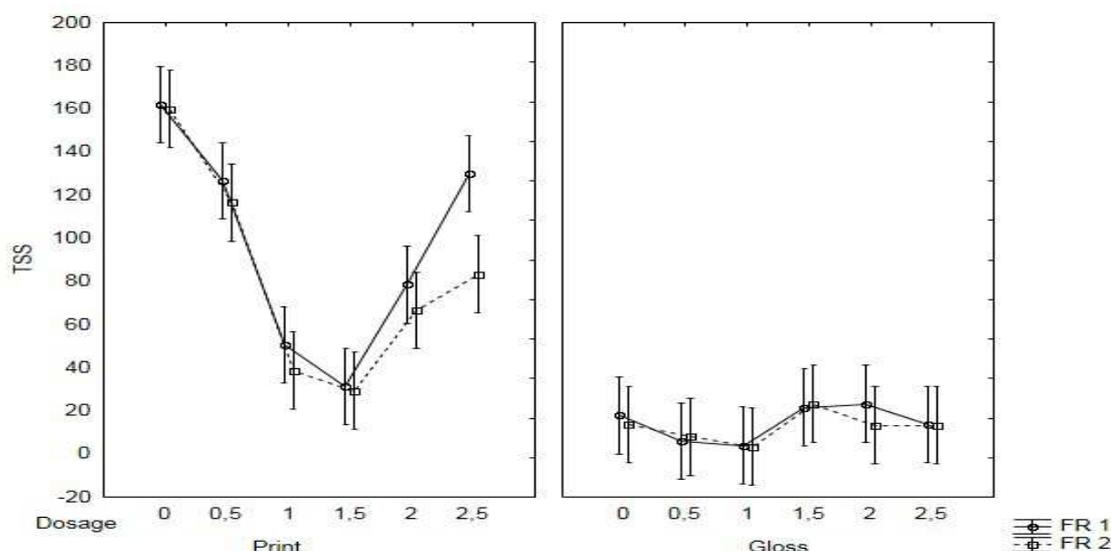


Fig. 4: Interaction between: dosage x flow rate x paper type for the TSS parameter.

Based on Figure 4 we can say that flow rates are statistically similar, except for the dose 2.5 mg/L in clear water from print paper.

The treatment of clear water from gloss paper had the best overall results in the removal of fibers (TSS) (Figure 4), the maximum removal value of 99% for a dosage of 1 mg/L, followed by 98% for the dose 0.5 mg/L, both can be considered statistically similar to all other dosages tested, which had efficiency above 94% (Table 6). Demonstrating residual amounts of up to 2 mg/L of TSS remaining, being highly efficient at removing TSS parameter. These results are similar to tests performed by Woollenet *al.* (1995), which reported the removal almost complete of existing suspended solids in a paper machine effluent using DAF followed by sand filter and disc filter.

The treatment of clear water from print paper also showed excellent TSS removal values with 94.6% (Table 5) for 1.5 mg/L dosage, being statistically the same as the dosage 1 mg/L (Figure 4) with 92.9%, reaching residual minimum value of 24 mg/L TSS remaining in the effluent collected for analysis. The lower value found

for TSS removed was in the case of DAF without adding cationic polymer, with efficiency of 70.6% (Table 5). These values are accepted within the range of 70-98%, as described by Lanet *et al.* (2009), as typical of removing suspended solids using DAF as a primary treatment, generally requiring the use of coagulants and flocculants to achieve high efficiencies of removal.

The clear water from gloss paper performed better in removing TSS parameter compared to the clear water from the print paper (Figure 4). One can attribute this result the basic composition of each paper, as the gloss paper has long fibers (2 to 5 mm) in its composition, which may have contributed to the formation of more dense flakes and easy flotation. The print paper is basically composed of short fibers (0.5 to 2 mm), which due to its size the formation and flotation of the flakes may have lower efficiency. The particles that pass through the flotation process must be in a particle size range ideal for presenting good recoveries. This range depends on the ideal material that will float, since the materials do not have the same characteristics (Gorczyca and Klassen, 2008; Moruzzi and Reali, 2014).

Mehta *et al.* (2003) conducted a research to optimize the removal of suspended solids and organic matter of paper machine effluent, particularly alkali, and reached a TSS residual of 34 mg/L using a dosage of 25 mg/L of high molecular weight cationic polymer. Dixit *et al.* (1991), reported that the addition of cationic polymer in the system is a technology typically used in paper machine. The cationic polymer is effective in the retention of fibers, organic matter and mineral fillers.

The recovery of paper fibers is a strategy to preserve forest resources and to save costs. A large number of studies have been published regarding the optimization of DAF parameters, using different coagulants and reusing treated water (Silva *et al.*, 1999; Constanzi and Daniel, 2002; Mehta *et al.*, 2003; Ben *et al.*, 2004; Pokhrel and Viraraghavan, 2004; Delgado-aguilar *et al.*, 2014), confirming the great interest in this field.

Optimal configuration:

In the treatment of clear water from print paper, the optimal polymer dosage was 1 mg/L, reaching removal efficiency values of 92.9%, 73%, 97% for TSS, apparent color, turbidity, respectively. Did not occur COD removal (Table 5).

In the treatment of clear water from gloss paper, the optimal polymer dosage also was 1 mg/L with removal of 99%, 97%, 98% and 48.5% for TSS, apparent color, turbidity and COD respectively (Table 6).

In general, for the studied parameters there was no statistically significant difference between the tested flow rates. Thus, in both cases, we can adopt the highest speed flow rate (FR 1 = 18 cm/min), which implies smaller reactors in industrial scale, making the system more compact.

Conclusion:

The dissolved air flotation, with the aid of cationic polymer, improved the characteristics of clear water, being recommended for primary treatment, to remove fibers lost in the production process. The total suspended solids, which represent the fiber contained in clear water were efficiently removed by DAF, reaching 99% of efficiency.

The use of cationic polymer allowed the formation of large, well-structured flakes, facilitating the flotation of the suspended solids. The cationic polymer used is indicated, as the industry already uses this product in the retention process in the formation of the sheet, it will require a small dosage of the product, which got good response at the natural pH of the effluent.

The optimal configuration determined by laboratory tests was the dosage of 1 mg/L of cationic polymer and flow rate of 18 cm/min for both types of paper.

DAF treatment of clear water from gloss paper obtained a reduction above 97% for all parameters studied, with the exception of COD with 48.5%.

DAF treatment of clear water from print paper reached removal efficiency values of 92.9% for TSS, 73% for apparent color, 97% for turbidity and had no COD removal.

Whereas industry produces these two types of paper alternately, according to demand, the treatment by DAF of both effluent produced on the paper machine would be feasible with the aid of cationic polymer.

With the possibility of a compact floater machine, low operating cost and energy efficient, DAF has proven to be especially suitable for applications in pulp and paper wastewater due to its high capacity hydraulic load and advanced performance, specifically being applied the recovery of suspended solids containing fibers, fines and fillers.

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