

Evaluation of the SuperBatch™ Pulping Process for *Pinus taeda*

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ABSTRACT in this research was evaluated the efficiency of the SuperBatch™ kraft pulping process in comparison to conventional pulping process as well as the effects of anthraquinone (AQ) aiming the production of unbleachable grades (kappa number 58 ± 2) for *Pinus taeda* with 9 years old, the main specie used in Brazil to produce long fiber pulp. A study of the impregnation phase in the conventional cooking was performed; the heating up time was variable and the time at the maximum temperature was adjusted to a H-factor of 2300. It was also studied the evolution of the delignification during the SuperBatch™ and conventional process. The pulping results show that in the SuperBatch™ process a smaller amount of rejects was produced than conventional process; the addition of anthraquinone was efficient to reduce the kappa number in the SuperBatch™ process, however without significant gains of selectivity; in the conventional process with the addition of AQ a reduction of 0,5 percentual points in the initial active alkali was possible. During the evaluation of the impregnation phase in the conventional process the adoption of a longer time during the heating up phase results in a reduction of the rejects and an increase in the screened yield. Finally, in the study of the delignification during the cooking, the logistic mathematical model developed showed a good adjustment with three phases identified for all the cases: (i) initial delignification phase when a small amount of lignin is removed, (ii) bulk delignification phase, when the intensity of delignification increases and most of the lignin is dissolved and (iii) residual delignification phase, when the delignification is small again. In the SuperBatch process during the impregnation phase, practically did not happen any reaction between reagents and lignin; during the hot black liquor (HBL) about 25-30% of the lignin is dissolved and in the bulk phase about 50-55%. The addition of AQ in the SuperBatch process increased the intensity of the reactions with the lignin, resulting in a faster delignification during the bulk delignification.

INTRODUCTION

Brazil has a prominent status as a pulp producer with a production about 11.000,000 ton in 2006 showing a competitive industry with a great potential of growing due to advanced silvicultural techniques, available areas to new plantations, suitable raw material for pulp and paper production, modern mills, capacitated human resources etc.

However, Brazil is an importer of long fibers instead of what happens regarding short fiber because Brazil is the main world producer of short bleached fiber of market pulp. About 80% of the national production is from eucalyptus trees and 20% from pinus species, especially *Pinus taeda* (loblolly pine). For this reason, researches to develop and increase the production of pulp from softwood are necessary, relevant and quite important to pulp and paper industry in Brazil.

Nowadays, there are about 1,8 millions hectares of pinus species planted in Brazil with special reference to *Pinus taeda*, which was introduced in Brazil in 1936 and is originally from southern USA^{1,2}. Brazil has developed many silvicultural techniques to improve the rate of growth and the quality of wood for many uses so that this specie is completely adapted to Brazilian conditions and its uses for the national industry.

In a tree, the walls of cells are structured in layers and the outermost amorphous and lignin rich layer, called the middle lamella glues cells together. The kraft pulping is based on the principle of fiber liberation by dissolving enough lignin from the middle lamella of wood that the fibers are freed. ³.

Since the discovery of the kraft pulp process a lot of developments have been tested and implemented to improve its efficiency both in quantitative and qualitative aspects. During the past decades, the main modifications have been directed to increase the yield of the process and the intensity of the delignification to implement ECF and TCF bleaching sequences⁴.

In the late 1970s and early 1980s, a concept for more selective kraft process – called modified kraft pulp – became available. This allowed the kraft pulping industry to respond to environmental challenges without sacrificing kraft pulp quality. Lower kappa numbers were therefore possible without loss in quality⁵.

During the 1980's a lot of studies were accomplished to develop new discontinuous kraft processes, that initially had the focus on reduce the energy spent. In comparison to continuous process, batch processes consume about the double of energy⁶.

These new batch pulping processes use liquor displacement to recover hot black liquor at the end of cooking allowing the reuse of the heat content in subsequent batches. In addition, trials to combine modified cooking chemistry (alkali profiling and low content of dissolved matters) with energy efficient liquor displacement had occurred and among the developments is the SuperBatch™ concept that involves both energy efficiency and efficient use of residual and fresh cooking chemicals⁵.

Another alternative that has been considered to modify the kraft process aiming the increase of the yield is the use of additives in the process that is an interesting option because the costs to installation are low⁷. Among the existences additives, specifically the anthraquinone (AQ) has shown very efficient.

Thus, this research aimed to study the delignification of *Pinus taeda* during the SuperBatch™ process in comparison to conventional kraft process. Furthermore, was evaluated the addition of anthraquinone in both processes.

EXPERIMENTAL

Raw material

The cooking experiments were performed on industrial loblolly pine (*Pinus taeda*) chips with 9 years old, planted in south Brazil, state of Paraná. The material was supplied by Klabin. For the kraft-AQ cooks a commercial grade of anthraquinone powder was used.

The chips were screened to eliminate the fines and over-size chips.

It was measured the basic density, chemical compound and morphological characteristics to evaluate the basic characteristics of the wood.

Cooking

The cooks were performed in a M/K digester with 2 vessels of 10L of capacity, recirculation pump and a heater exchanger. To SuperBatch™ cooks were necessary to implement some modifications on the digester to be possible make the displacement of the liquors. In this case, one vessel was used as an accumulator of liquor and other one to perform the cook; it was installed two metering pump, one to inject the liquor from the accumulator vessel to the cooking vessel and other to extract the liquor from the cooking vessel. All the displacement of the cooking was controlled by on-line controls developed at the University of São Paulo (USP)

The conditions for the conventional cooks are described on Table 1.

Table 1. Conventional pulping parameters.

Parameter	Value
Active alkali (as Na ₂ O, % on o.d. wood)	*
Sulfidity (% on o.d. wood)	25
Liquor/wood ratio	5
Maximum temperature (°C)	170
Heating-up time (minutes)	90
Cooking time (minutes)	120
AQ charge (% on o.d. wood)	0,10
H-factor	2300

* AA charge variable to reach the kappa number specified.

The active alkali applied on conventional cooking was variable to reach the kappa number of 58±2. When added anthraquinone in the process, the active alkali applied was changed in order to keep the kappa number in the range determined.

The conditions to SuperBatch™ cooking are described on Table 2 and 3.

Table 2. SuperBatch™ pulping parameters

Parameter	Value
Total active alkali (as Na ₂ O)	22
Liquor/wood ratio	5
AQ charge (% on o.d. wood)	0,10
H-factor	1037

Table 3. SuperBatch™ pulping parameters in each phase.

Parameter	WBL	HBL	Cooking	Washing
AA split (%)	25	35	40	-
Sulfidity (%)	50	40	25	-
Temperat. (°C)	90	150	165	80
P1 (minutes)	0	37	15	15
P2 (minutes)	65	47	70	45

WBL = Warm black liquor; HBL = Hot black liquor

P1 = phase of displacement of liquors

P2 = phase at the conditions established

In the SuperBatch™ cooking, the AQ charge was added in the beginning of the cook. In addition, no changes in the total active alkali charge was made in order to keep the kappa number specified, but just was observed its impacts on the process.

The profile “time x temperature” based on Table 3 to the SuperBatch™ process can be seen in Figure 1.

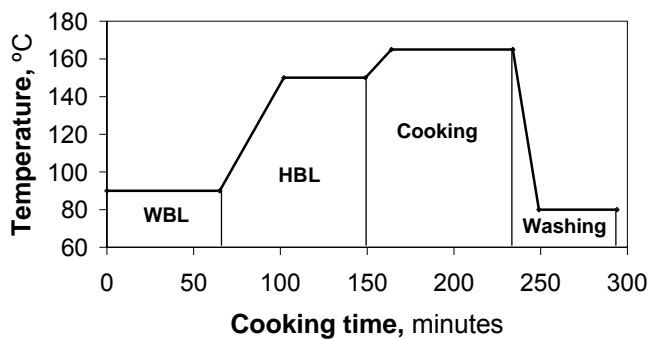


Figure 1. Profile time x temperature to SuperBatch™

Heating up trials

In conventional cooking was studied the influence of the heating up time due to its importance in the whole process.

Thus, the heating up time was variable in the following times: 45, 60, 75, 90, 105, 120 and 135 minutes and the time at the maximum temperature (170°C) was adjusted to keep the H-factor constant in 2300. The other parameters of cooking are equal to Table 1.

Delignification during the pulping

In conventional process, the heating up time that showed the best results, with special reference to the unscreened yield, was made a study of the delignification during the pulping. For this study, the cook was interrupted in each 15 minutes and samples of pulp and liquor were taken.

For the SuperBatch™ process, a similar study was carried out, stopping the process in each 15 minutes either.

TAPPI test methods were used for all pulp evaluations, except where otherwise noted.

RESULTS AND DISCUSSION

Information on the basic characteristics of the raw material used for pulping is show on Table 4.

Table 4. Raw material characteristics.

Parameter	Value	S.D.
Basic density (g/cm³)	0,423	0,013
Extractives content (%)	4,50	0,49
Lignin content, (%)	28,40	0,49
Holocelullose content, (%)	67,10	0,96
Fiber length, (mm)	4,22	0,85
Fiber width, (μm)	48,53	11,05
Lumen diameter, (μm)	32,20	13,52
Wall thickness, (μm)	8,16	3,13
Wall fraction, (%)	34	-

S.D. = Standard Deviation

The results presented on Table 4 show that the material used on this research can be considered representative for the specie *Pinus taeda* for pulp production in Brazil

Table 5. Parameters of SuperBatch™ and conventional cooking.

Parameter	SuperBatch™		Conventional	
AQ charge (%)	0,00	0,10	0,00	0,10
AA charge (%)	22,0	22,0	14,5	14,0
H-factor	1037	1037	2300	2300
Kappa number	56,80	52,75	56,20	57,00
Unscreened yield (%)	57,79	54,15	55,38	56,74
Screened yield (%)	52,31	48,35	43,34	43,09
Rejects (%)	5,48	5,80	12,05	13,65
Selectivity	1,017	1,027	0,985	0,995

Table 6. Consumption of active alkali.

Process	Kraft		Kraft-AQ	
	%	g	%	g
SuperBatch™				
Warm Black Liquor	7,0	19,9	5,2	14,9
Hot Black Liquor	24,8	70,8	21,8	62,4
Cooking	19,6	56,1	23,3	65,8
Total	51,4	146,8	50,4	144,1
Conventional				
Cooking	73,6	138,8	73,1	135,5

The SuperBatch™ process requires a higher amount of active alkali. This higher demand for alkali in modified cooking has been observed by other authors. This difference occurs fundamentally for the necessity of distribution of alkali during the modified cooking; nevertheless, this not means a higher consumption of active alkali as can be seen on Table 6, where the amount of active alkali consumed in grammas on SuperBatch™ and conventional cooking is practically the same.

The H-factor has been quite different as well; while in the SuperBatch™ process the value was 1037 on conventional process it was 2300, showing a great saving of energy.

According to Hakamaki; Kovasin (1991)⁶ the H-factor demand for a given kappa number level in conventional cooking is higher than that in SuperBatch™ cooking and the phase of hot black liquor (HBL) has a fundamental role. These authors showed that as high the temperature in this phase as low will be the H-factor required because the speed of delignification is increased, thus rendering selectivity and reducing cooking time for a given kappa number – the maximum temperature studied was 145°C.

The kappa number in the conventional cooking is practically the same between kraft and kraft-AQ process because with the addition of anthraquinone was possible to reduce the AA applied from 14,5% to 14,0%. In the SuperBatch™ cooking, where no changes were made, it was observed a reduction of about 4 points, confirming its effects on the degradation of lignin and the procedure to apply AQ in the beginning of the cooking. Besides, in case to keep the kappa number on the range specified, the addition of anthraquinone allows changes on the AA charge, time and/or temperature.

SuperBatch™ cooking without AQ, shows a greater unscreened yield than conventional cooking (without and with AQ), but when is added AQ in the SuperBatch™ cooking the unscreened yield drops in 3,5 points, however, the yield is also related with the amount of lignin in the pulp and considering that SuperBatch™-AQ resulted in a lower kappa number, it is possible to conclude that the lower amount of lignin justify partially this lower yield.

According to McDonough⁸, with reference to the manufacture of pulps for unbleachable grades, where retention of lignin in the pulp fibers is less objectionable than in the case of bleachable grades, lignin retention is desirable from the yield standpoint and allowable to the extent that it can be obtained without having too great an adverse effect on pulp properties. Thereby, with the addition of AQ is possible to change the initial parameters of SuperBatch™ cooking such as the active alkali so that the kappa number will keep in the range specified and the likely consequences is the increasing of the unscreened yield due to the higher lignin content and retention of carbohydrates with the lower AA charge.

The amount of rejects is too higher in conventional cooking in comparison to SuperBatch™ cooking, this benefit of the SuperBatch™ cooking is due to the best impregnation promoted during the WBL phase. Weckroth; Hiljanen⁹ observed the same effects on their research. On table 6 is possible to see that the consumption of alkali during the WBL phase is very low, corroborating that this phase has not the aim to promote reaction between wood and chemicals, but it has the objective of heat up the chips and promote their impregnation.

The selectivity, calculated dividing unscreened yield by the kappa number, is a little higher on SuperBatch™ cooking than conventional cooking and the addition of AQ increased the selectivity on few units.

Heating up (conventional pulping)

A fast heating up of the chips and liquor on conventional cooking can promoted an uneven delignification into the chips by the kraft liquor, on the other hand, a very long time to the heating up period could cause a great degradation

of the carbohydrates render on yield losses and lower production rate. Thus, this part of the research aimed to determine, among the heating up times studied, what is that one that results on the best performance of the process, with special reference to unscreened yield.

For both kraft and kraft-AQ process, on Figure 2 can be seen that initially there is a tendency to increase the unscreened yield and then the yield starts to drop. This behavior is more pronounced on kraft-AQ process.

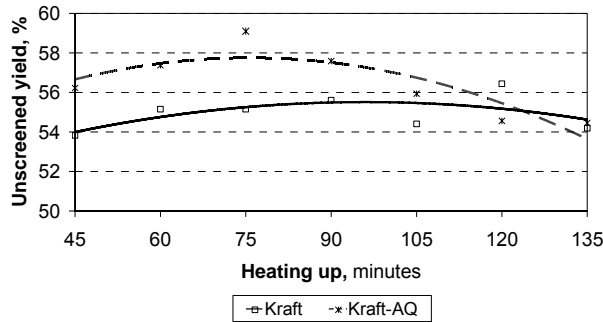


Figure 2. Unscreened yield.

A short heating up time could cause deficiencies on the impregnation of the chips, what can be confirmed on Figure 3, where the rejects amount is higher on the shortest time and it is getting smaller with longer heating up times.

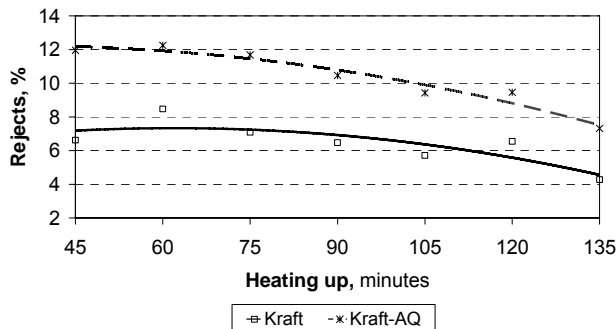


Figure 3. Rejects.

The heating up of the chips without a good impregnation by the cooking liquor promotes acid hydrolysis so that a higher amount of carbohydrates is degraded resulting in a lower screened yield as can be seen on Figure 4.

On the highest heating up times the lower unscreened yield is related mainly to the drop of rejects. Thereby, there is a middle point where is observed a greater unscreened yield, when the degradation of carbohydrates is not so intensive and the reduction of rejects do not affect excessively the unscreened yield.

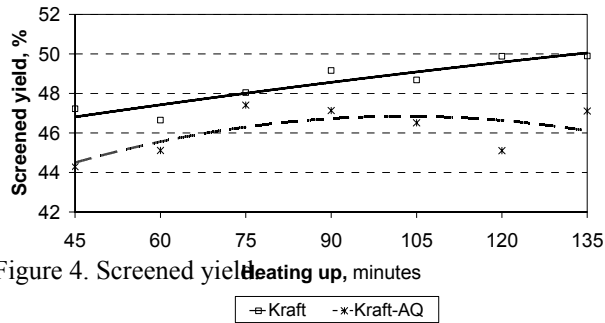


Figure 4. Screened yield vs heating up, minutes

Thus, with special reference to unscreened yield, the best performance was obtained with a heating up time of 90 minutes for kraft process and 75 minutes for kraft-AQ process.

Delignification during the pulping

Established the parameters of SuperBatch™ and conventional cooking, it was done a study about the delignification of the wood during the pulping processes.

Using the data of percentage of lignin on wood versus the cooking time (expressed in percentage, %), it was adjusted a mathematical logistic model:

$$\text{Lignin} = (100-h) \frac{\alpha e^T}{1+\alpha e^T} + h \quad (1)$$

The logistic model fitted well for all data and the parameters estimation are show on Table 7.

Table 7. Parameters estimation.

Process		α		H
SuperBatch™	kraft	1083,2	-0,1248	11,8391
SuperBatch™	kraft-AQ	169,6	-1001	14,1328
Conventional	kraft	65,7	-0,0865	15,8277
Conventional	kraft-AQ	313,5	-0,1269	20,9382

Based on logistic model and the parameters estimation, on Figure 5 and 6 there were the delignification of SuperBatch™ and conventional cooking during the kraft pulping respectively.

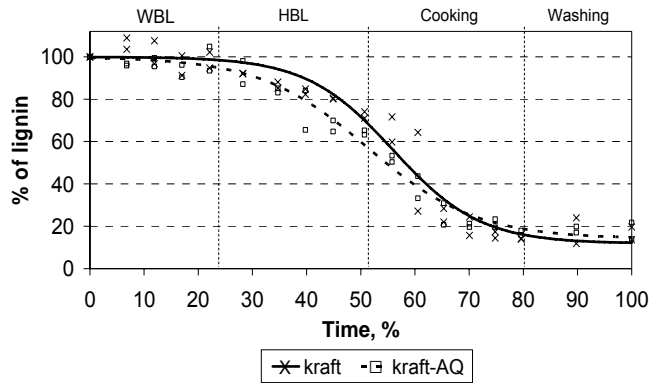


Figure 5. Amount of residual lignin (wood based) - SuperBatch.

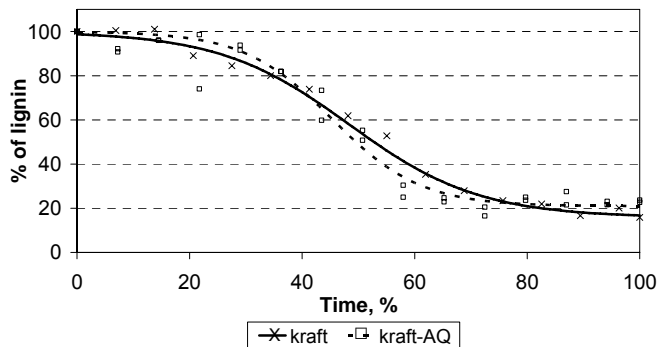


Figure 6. Amount of residual lignin (wood based) - conventional.

Many authors have already described a similar behavior of delignification, but in most cases studying the conventional cooking. It is possible to see three phases: initial phase when a small amount of lignin is removed; bulk phase when practically most of the lignin is removed and finally the residual delignification where the delignification is quite small.

During the warm black liquor phase (WBL) practically there is no removal of lignin due to the low temperature and AA charge, thereby in accordance with Table 6, where it shows that the consumption of alkali is very low, it shows that this phase has the main objective of impregnate the wood with the kraft liquor. On the hot black liquor phase (HBL) the temperature is heated up from 90°C to 150°C and 35% of the total alkali charge is added in the process so that the rate of delignification is higher and about 25-30% of the lignin is removed. The cooking phase removes most of the lignin from the wood (about 50-55%) because it is used the highest temperature of the whole process and the amount of reagents available is also the highest. Finally, the washing phase did not remove any significant amount of lignin and this phase even does not have this objective.

It was observed that the fiber liberation point had occurred at 65% of the total pulping time, when there is present 25% of the initial amount of lignin and the unscreened yield is about 60%.

The delignification behavior of conventional cooking is quite similar than SuperBatch™ cooking, where it is possible

identify the initial, bulk and residual delignification phase. During the initial phase about 20% of the lignin is removed from the wood and practically all the rest of delignification had occurred during the bulk delignification. The delignification in the residual phase is despicable.

The residual phase on the conventional cooking occurs for about 30% of the total cooking time as well as on SuperBatch™ cooking. However, on the conventional process the whole residual phase occurs at 170°C, while on SuperBatch™ cooking the residual phase under the maximum temperature is small and most of the residual delignification is during the washing phase, where the alkali charge and temperature are low. Thus, as the residual delignification under the more aggressive conditions is shorter than in conventional cooking, the loss of carbohydrates could also be smaller, once during the residual delignification the removal of lignin is small and the loss of carbohydrates could be great due to drastic conditions.

The fiber liberation point on conventional cooking had occurred at 85% of the total pulping time. At this moment there is about 20-25% of lignin and an unscreened yield of 55%. Thus, in comparison to SuperBatch™ cooking, the conventional cooking takes more time to start the fiber liberation point and when it happens the yield is lower at a similar amount of lignin. Probably these results are due to the better impregnation by the SuperBatch™ cooking, which allows the chemicals to reach easily to the middle lamella promoting a better individualization of the tracheids; as a result, the SuperBatch™ cooking showed a lower amount of rejects.

Despite the low amount of lignin removed during the residual phase, the continuous of the cooking to both pulping process is important to decrease the amount of rejects because was observed a substantial reduction of this parameter in each point of sampling. Therefore, the little amount of lignin removed during this phase probably is located on middle lamella. Thus, the extension of the cooking time, could be studied aiming the reduction of rejects, but a special attention to yield should be noted.

The AQ showed a catalytic effect on SuperBatch™ cooking because the rate of delignification during the bulk phase was higher.

CONCLUSIONS

The SuperBatch™ cooking is a suitable technology for the pulp production from *Pinus taeda* in Brazil;

The active alkali charge is about 55% higher on SuperBatch™ cooking than on conventional cooking.

The H-factor is 45% lower on SuperBatch™ cooking than on conventional cooking to the conditions studied;

SuperBatch™ cooking resulted in a substantial reduction of the rejects probably due to its better impregnation and the application of AQ in this process promoted a reduction of 3,5 points on kappa number, however without gains in selectivity ;

SuperBatch™ cooking was a little bit more selective than conventional cooking;

The mathematic logistic model fitted well for all process studied;

Three delignification phases were identified to both pulping process: initial, bulk and residual delignification;

On the SuperBatch™ cooking during the warm black liquor phase (WBL) the delignification is practically inexistence and the most delignification occurs during the hot black liquor (HBL) and cooking phase;

Anthraquinone accelerated the delignification in the bulk phase on SuperBatch™ cooking;

The study of the heating up phase on conventional cooking showed that longer heating up times resulted in a reduction of rejects and increase in screened yield

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Evaluation of the SuperBatch™ Pulping

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Introduction

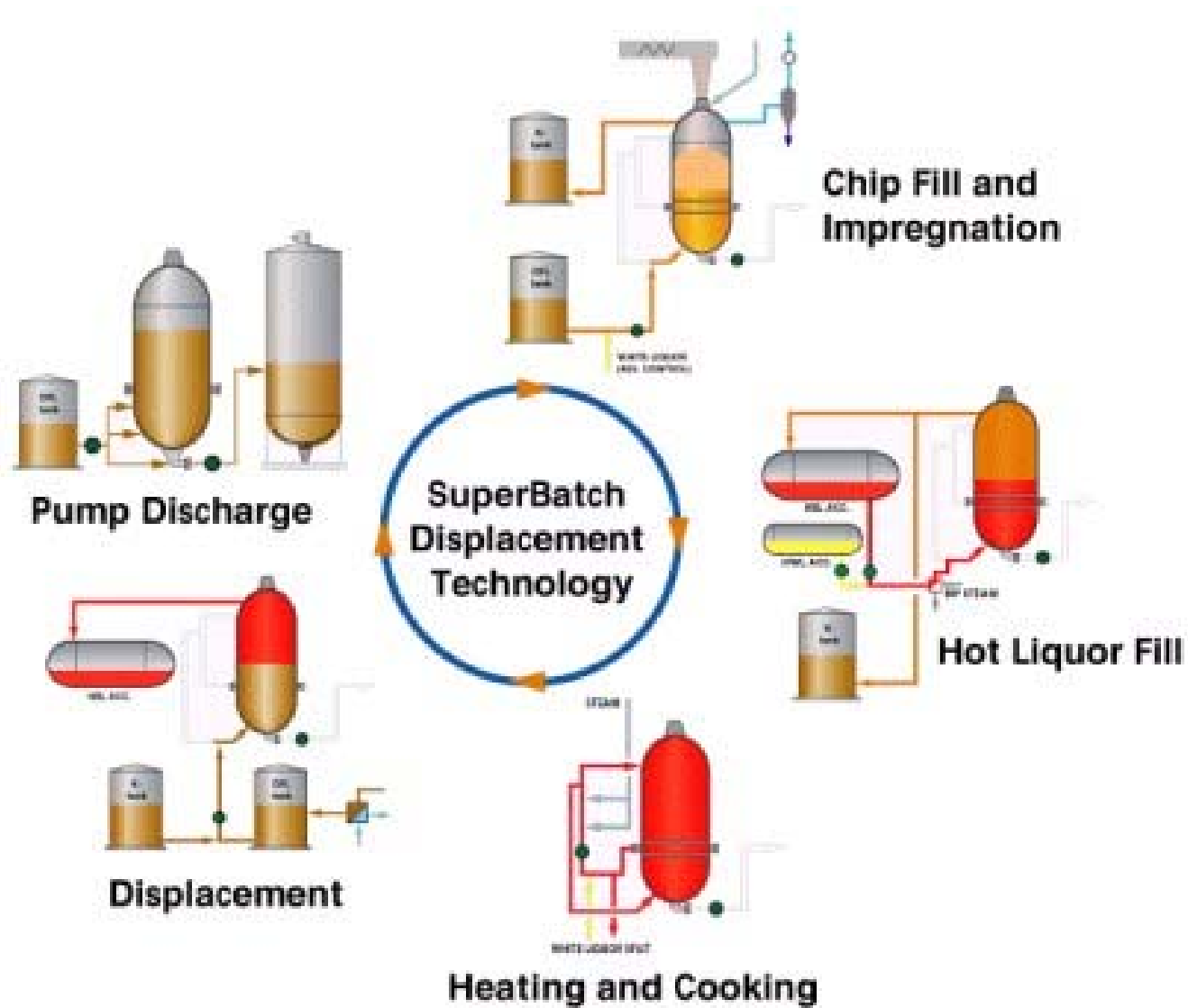
- Developing modified batch cooking
 - Improving energy and chemical utilization
- Anthraquinone

Introduction

This research aimed to study the delignification of *Pinus taeda* during the SuperBatch™ process in comparison to conventional kraft process.

Furthermore, was evaluated the addition of anthraquinone in both processes.

SuperBatch™ Pulping



Experimental

- **Raw material**

- Industrial loblolly pine (*Pinus taeda*) chips with 9 years old,
- planted in south Brazil, state of Paraná, supplied by Klabin.
- For the kraft-AQ cooks a commercial grade of anthraquinone powder was used.
- It was measured the basic density, chemical compound and morphological characteristics to evaluate the basic characteristics of the wood.

Experimental

- **Cooking**

Kappa number target of 58

SuperBatch™ pulping parameters

Parameter	Value
Total active alkali (as Na ₂ O)	22
Liquor/wood ratio	5
AQ charge (% on o.d. wood)	0,10
H-factor	1037

SuperBatch™ pulping parameters in each phase.

Parameter	WBL	HBL	Cooking	Washing
AA split (%)	25	35	40	-
Sulfidity (%)	50	40	25	-
Temperat. (°C)	90	150	165	80
P1 (minutes)	0	37	15	15
P2 (minutes)	65	47	70	45

Experimental

- **Cooking**

Conventional pulping parameters

Parameter	Value
Active alkali (as Na ₂ O, % on o.d. wood)	*
Sulfidity (% on o.d. wood)	25
Liquor/wood ratio	5
Maximum temperature (°C)	170
Heating-up time (minutes)	90
Cooking time (minutes)	120
AQ charge (% on o.d. wood)	0,10
H-factor	2300

* AA charge variable to reach the kappa number specified

Experimental

the heating up time was variable in the following times: 45, 60, 75, 90, 105, 120 and 135 minutes and the time at the maximum temperature (170°C) was adjusted to keep the H-factor constant in 2300. The other parameters of cooking are equal to Table 1.

- **Heating up trials (conventional cooking)**

- Heating up time was variable: 45, 60, 75, 90, 105, 120, 135 min.;

- H-factor constant: 2300

- Time at the maximum temperature was adjusted to keep the H-factor constant at 2300

- Maximum temperature constant at 170°C;

- Others cooking parameters were the same

Experimental

- **Delignification during the pulping**

In conventional process, the heating up time that showed the best results, with special reference to the unscreened yield, was made a study of the delignification during the pulping.

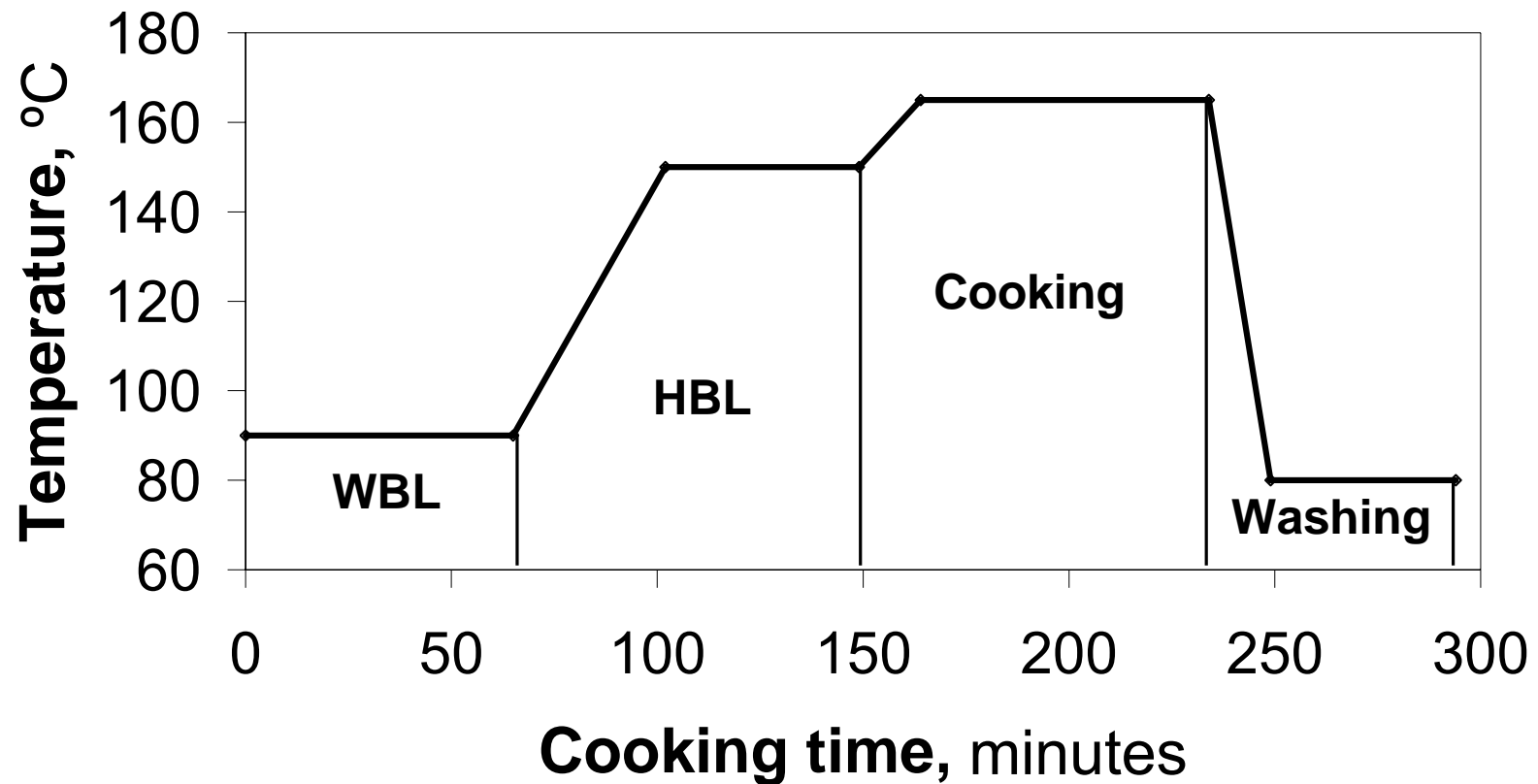
For this study, the cook was interrupted in each 15 minutes and samples of pulp and liquor were taken.

For the SuperBatch™ process, a similar study was carried out, stopping the process in each 15 minutes either.

TAPPI test methods were used for all pulp evaluations

Experimental

- SuperBatch™ pulping profile – time x temperature



Results

- Raw material characteristics

Parameter	Value	S.D.
Basic density (g/cm³)	0,423	0,013
Extractives content (%)	4,50	0,49
Lignin content, (%)	28,40	0,49
Holocelullose content, (%)	67,10	0,96
Fiber length, (mm)	4,22	0,85
Fiber width, (μm)	48,53	11,05
Lumen diameter, (μm)	32,20	13,52
Wall thickness, (μm)	8,16	3,13
Wall fraction, (%)	34	-

S.D. = Standard Deviation

Results

- Parameters of SuperBatch™ and conventional cooking

Parameter	SuperBatch™		Conventional	
AQ charge (%)	0,00	0,10	0,00	0,10
AA charge (%)	22,0	22,0	14,5	14,0
H-factor	1037	1037	2300	2300
Kappa number	56,80	52,75	56,20	57,00
Unscreened yield (%)	57,79	54,15	55,38	56,74
Screened yield (%)	52,31	48,35	43,34	43,09
Rejects (%)	5,48	5,80	12,05	13,65
Selectivity	1,017	1,027	0,985	0,995

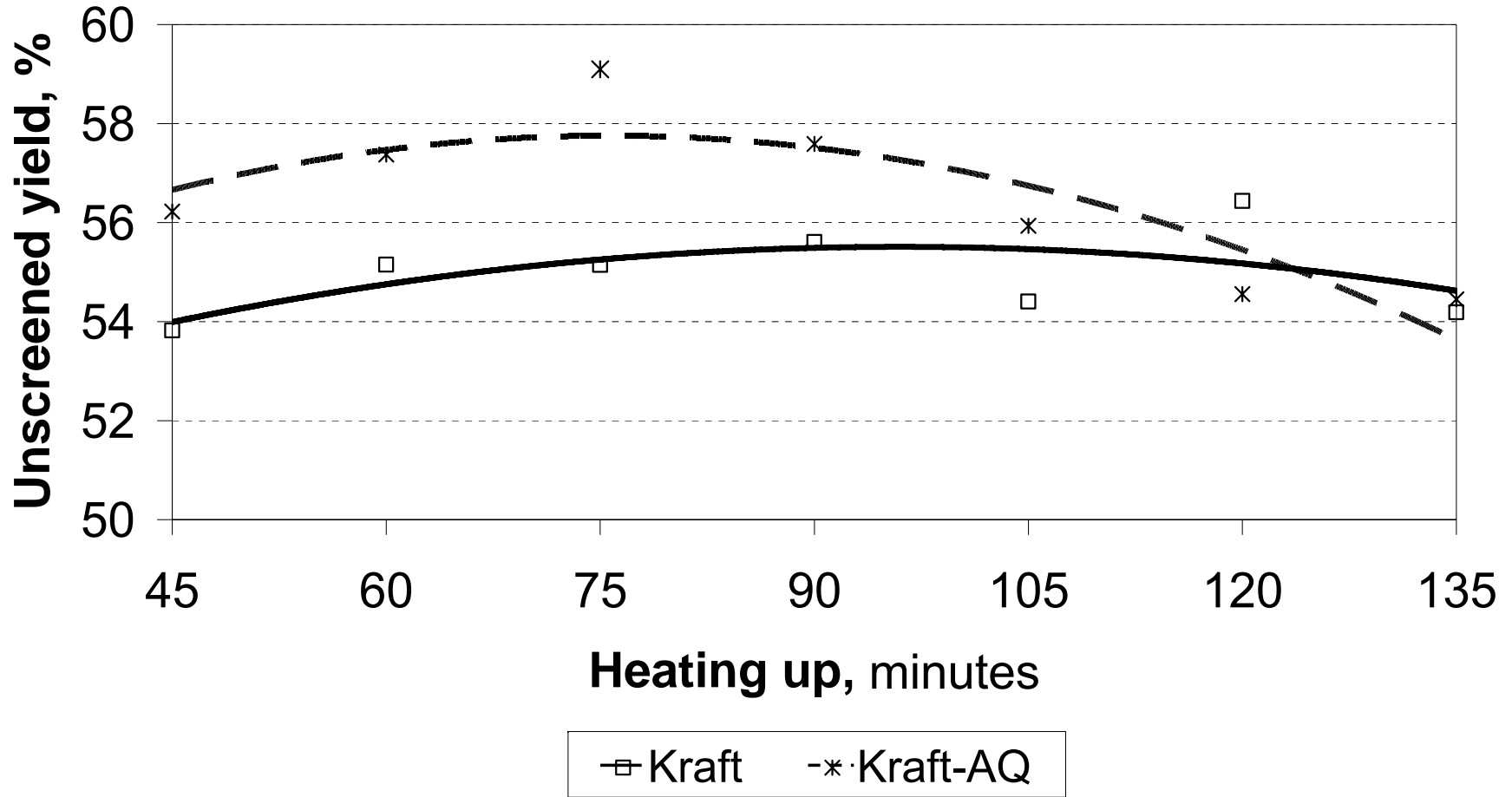
Results

- Consumption of active alkali

Process	Kraft		Kraft-AQ	
SuperBatch™	%	g	%	g
Warm Black Liquor	7,0	19,9	5,2	14,9
Hot Black Liquor	24,8	70,8	21,8	62,4
Cooking	19,6	56,1	23,3	65,8
Total	51,4	146,8	50,4	144,1
Process	Kraft		Kraft-AQ	
Conventional	%	g	%	g
Cooking	73,6	138,8	73,1	135,5

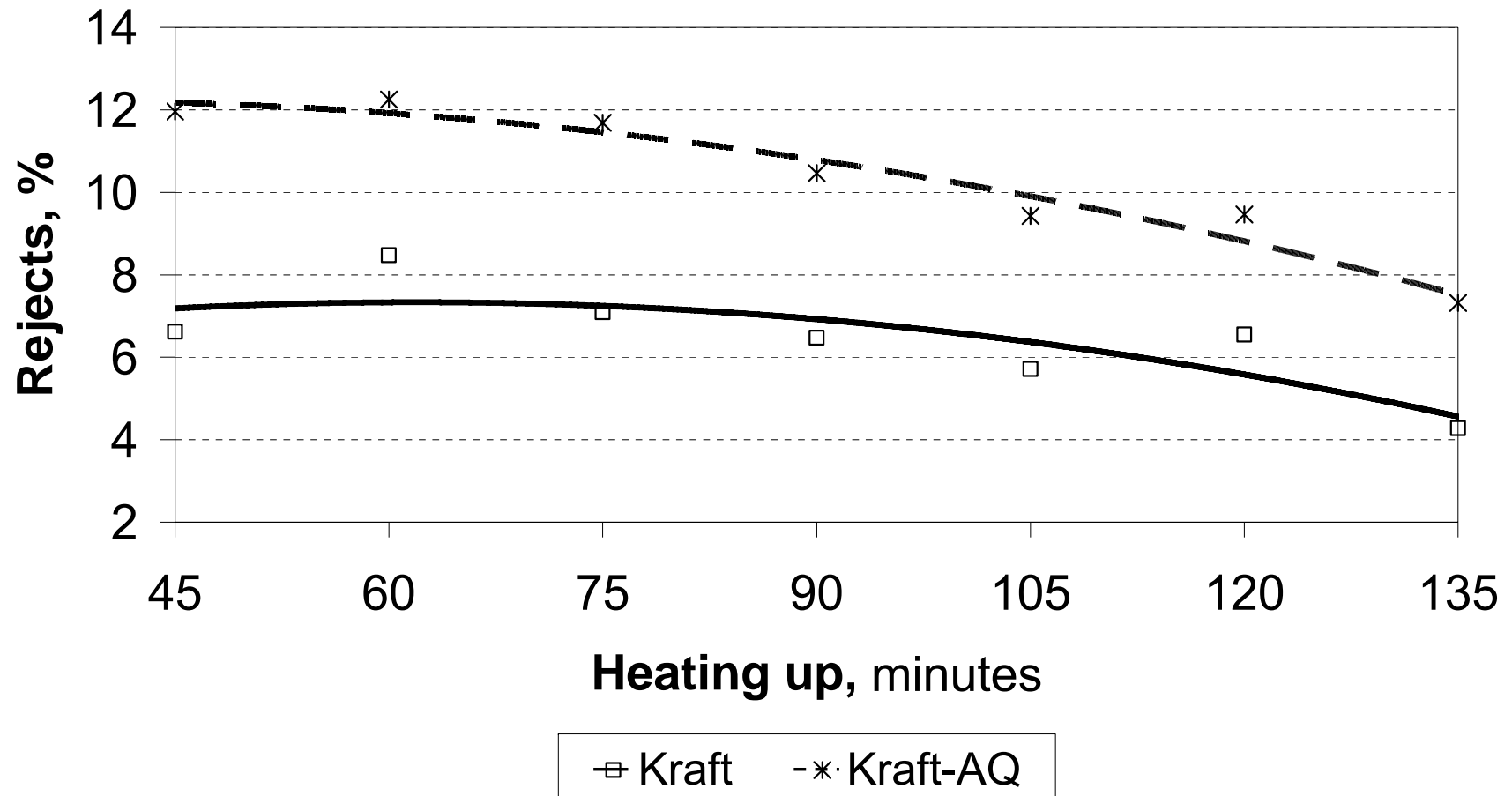
Results

- Heating up (conventional pulping)



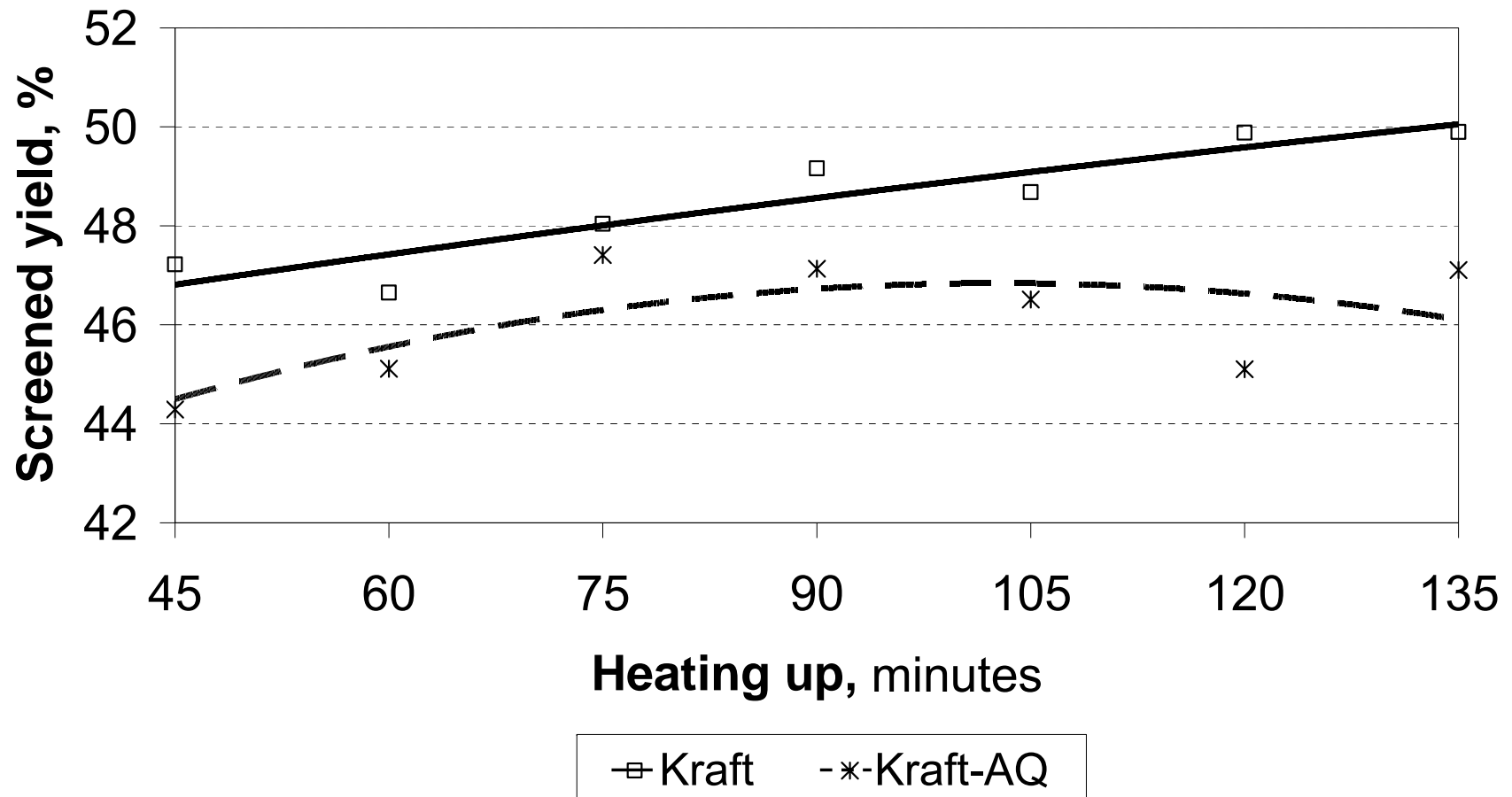
Results

- Heating up (conventional pulping)



Results

- Heating up (conventional pulping)



Results

- **Mathematical logistic model**

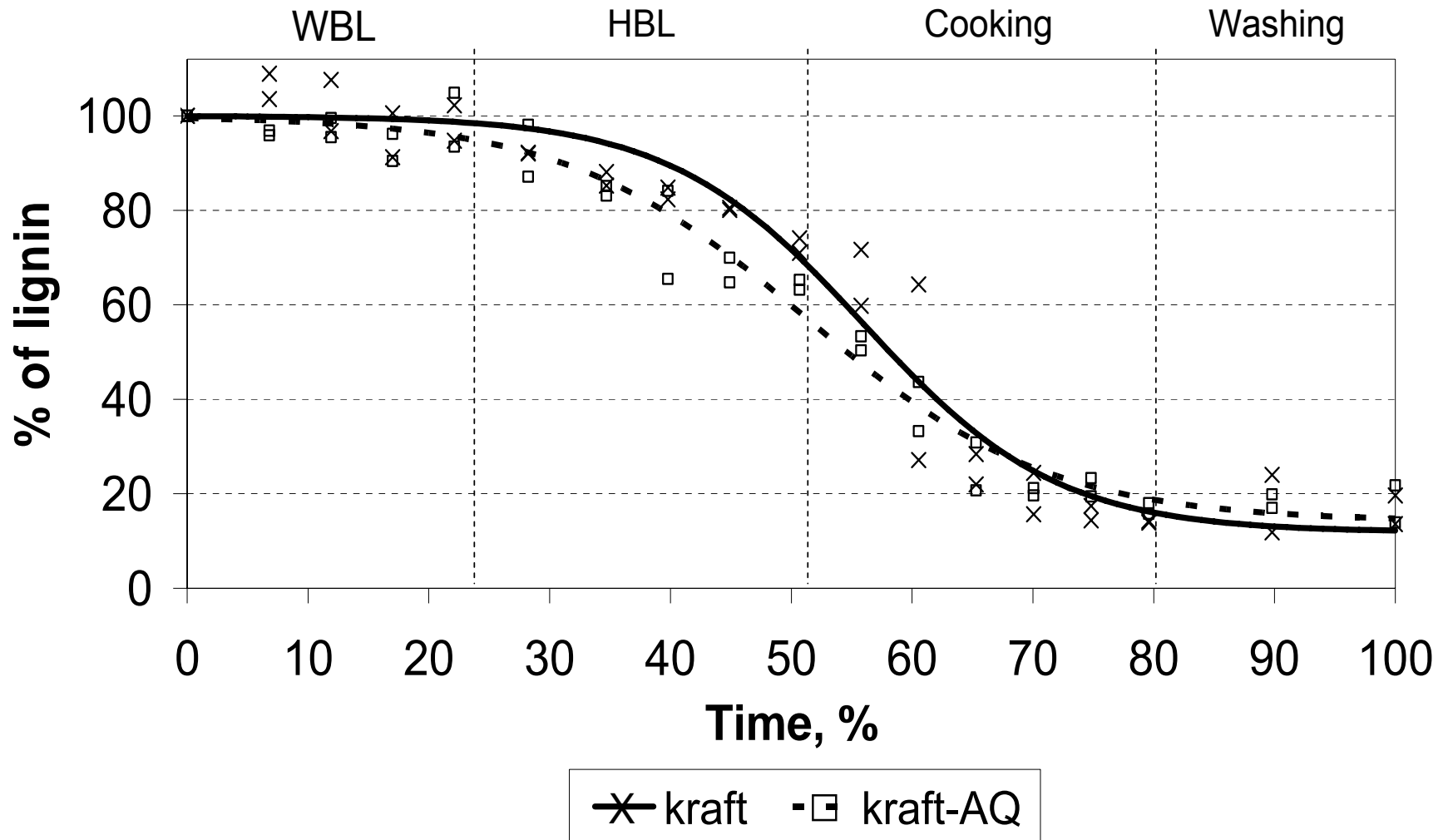
$$\text{Lignin} = (100-h) \frac{\alpha e^{\beta T} + h}{1 + \alpha e^{\beta T}} \quad (1)$$

- **Parameters estimation**

Process		α	β	H
SuperBatch™	kraft	1083,2	-0,1248	11,8391
SuperBatch™	kraft-AQ	169,6	-1001	14,1328
Conventional	kraft	65,7	-0,0865	15,8277
Conventional	kraft-AQ	313,5	-0,1269	20,9382

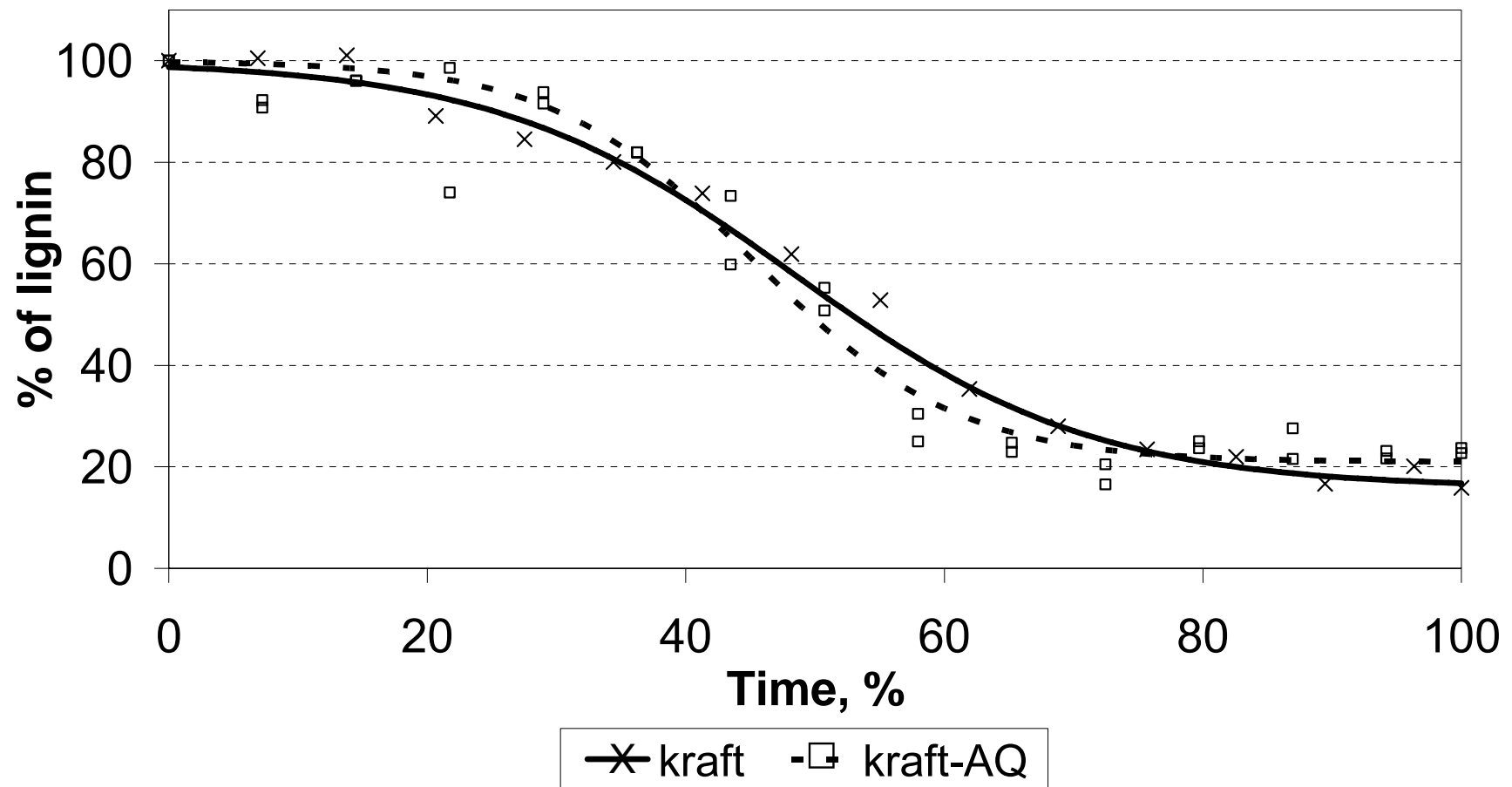
Results

• SuperBatch™ Pulping



Results

- Conventional cook



Conclusions

- The SuperBatch™ cooking is a suitable technology for the pulp production from *Pinus taeda* in Brazil;
- The active alkali charge is about 55% higher on SuperBatch™ cooking than on conventional cooking.
- The H-factor is 45% lower on SuperBatch™ cooking than on conventional cooking to the conditions studied;
- SuperBatch™ cooking resulted in a substantial reduction of the rejects probably due to its better impregnation and the application of AQ in this process promoted a reduction of 3,5 points on kappa number, however without gains in selectivity ;

Acknowledges

- Klabin S.A. for the wood supplied
- FAPESP – The State of São Paulo Research Foundation for the scholarship