



Impact of Storage Conditions on the Aging Rate and Brightness Reversion of Dissolving Pulp: The Role of Temperature and pH

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Abstract. This study examines how cellulose aging affects the degradation of dissolving pulp quality during storage. Dissolving pulp, with a consistency of 10%, is stored in tanks before being processed into pulp sheets. Over time, the quality of this pulp deteriorates, a phenomenon known as pulp aging. Key factors such as temperature and pH impact the rate of aging. To simulate this process, samples of 10% consistency pulp are placed in a water bath under storage-like conditions for 144 hours, with pH levels adjusted to 4, 6, and 8, and temperatures set at 50°C, 60°C, and 70°C. The quality metrics analyzed include brightness and viscosity, assessed every 24 hours. Findings indicate that the optimal storage conditions are at temperature 50°C and pH 8, where the aging process and degradation in quality are slower. Under these conditions, the rate of aging is up to three times slower compared to the highest rate, which occurs at pH 4 and 70°C. These results provide valuable insights for optimizing storage conditions to preserve dissolving slush pulp quality.

Keywords: Cellulose depolymerization, Pulp aging kinetic rate, Pulp storage parameter, Dissolving pulp, Cellulose degradation.

1 INTRODUCTION

Dissolving pulp is derived from raw materials such as wood, cotton, bamboo, chemical pulp, and other plant biomass. Dissolving pulp is a valuable commodity that generates substantial revenue for the global pulp industry [1]. The high cost of using cotton as a textile raw material, combined with the declining demand for traditional paper products, has led to a shift towards using pulp for paper production to textile production. Some products that utilize dissolving pulp as raw materials include viscose fibers, lyocell, and micro- crystalline cellulose (MCC), which are used in applications within the food, pharmaceutical, and paint and coating industries [2]. Compared to kraft pulp, dissolving pulp is characterized by a high cellulose content, often referred to as alpha-cellulose, exceeding 90%, high brightness levels, low macromolecular polydispersity index (PDI), and hemicellulose content <5% [3]. The market for dissolving pulp is projected to keep growing through 2020 and beyond. The steady growth in demand will attract more paper-grade pulp producers to shift over to the production of dissolving pulp [4].

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In 2021, worldwide pulp production (including dissolving pulp) exceeded to 190,000 million tons annually with the increment around 10,000 million tons from previous year. In 2020, Indonesia ranked as the 8th largest pulp producer worldwide, and by 2021, it advanced to the 7th position globally [5], [6]. Figure 1 shows the top ten wood pulp producers worldwide in 2021. Indonesia ranked 7th in wood pulp production globally after Finland with the total production exceeded 10,000 million tons. Indonesia has significant potential for growth in wood pulp production due to its abundant raw material resources, particularly its extensive wood resources. Figure 2 shows annual dissolving pulp export in Indonesia from 2017 until 2021 [7]. The annual growth in Indonesia's dissolving pulp exports highlights the significant potential for expanding dissolving pulp production in the country.

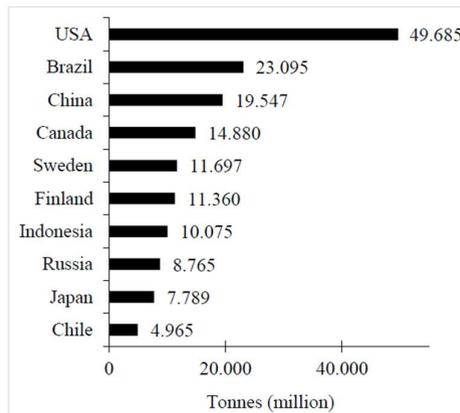


Fig. 1. Global wood pulp production rankings for 2021.

Dissolving pulp is produced from wood or cotton linters and characterized by its high cellulose content (over 90%). It possesses unique attributes including high purity, consistent molecular weight distribution, exceptional brightness, and high cellulose reactivity [8]. Dissolving pulp gets its name from its ability to dissolve in solvents or be converted into a uniform solution. This makes it a key raw material for producing textile fibers and cellulose derivatives, including cellulose esters, cellulose ethers, and other related compounds [9]. Up to 90% of dissolving pulp is produced from wood by a low yield (30–35% of wood) pulping processes such as sulfite and PHK pulping [4].

The PHK process allows for the use of both softwoods and hardwoods as raw materials due to its reduced sensitivity to wood's extractive content. Hemi-celluloses are removed during the initial pre-hydrolysis stage, followed by kraft cooking and a multi-stage bleaching process to produce high-purity dissolving pulp. While the PHK process provides advantages such as flexibility in raw materials, enhanced chemical recovery, and high-quality pulp, it also presents environmental challenges, notably significant water pollution due to the intensive bleaching requirements [2]. Additionally, the process has limitations, including increased production time due to the extra pre-hydrolysis steps (with total reaction times of 160–200 minutes for conventional kraft pulping and 240–270 minutes for PHK pulping), a lower yield of

dissolving pulp, averaging 38% compared to 48% for conventional kraft pulping, higher capital, chemical costs as more extensive pulping, higher inventory and storage space needs [4].

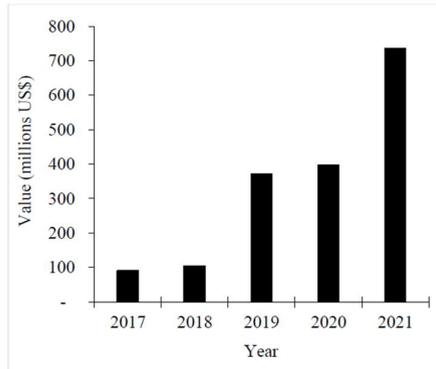


Fig. 2. Indonesia dissolving pulp export over year.

Dissolving pulp is used to produce regenerated fibers and cellulose derivatives, which have a significantly higher market value than conventional paper [10]. As a result, the standards for dissolving pulp are more rigorous, with properties specifically tailored for various applications. These characteristics are summarized in Table 3 [2]. Different applications require unique pulping attributes, highlighting the need for precise and targeted specifications. Dissolving pulp that has been bleached and meets quality requirements will be stored in a tank, typically at 10% consistency, before being processed into pulp sheets. This process facilitates storage and transportation for sale to customers. Dissolving pulp stored in tanks as a slurry will naturally age, with cellulose chains degrading. Consequently, some of the quality may deteriorate, causing it to no longer meet the required standards.

Cellulose degradation is an inevitable consequence of natural aging. There are two primary mechanisms of cellulose aging: hydrolysis and oxidation, which can occur separately or in combination. The aging of cellulose largely depends on the environment in which the pulp has been stored, the conditions it has been exposed to, and the stressors it has encountered. Key factors influencing cellulose degradation during natural aging include temperature, irradiation (UV/VIS, beta- or gamma irradiation), relative humidity or moisture content, exposure to various chemicals, presence of acids, metal ions, and fluctuations in environmental conditions [11].

Despite the diverse causes, their primary effect is remarkably straightforward: cellulosic hydroxyl groups are converted either into carbonyl structures (such as keto groups at C-2 or C-3, or aldehyde groups at C-6) or carboxyl structures (which occur only at C-6 and at the reducing end). The subsequent loss of molecular weight, or cellulose chain cleavage, is a later consequence of cellulose degradation [12]. Since cellulose aging is unavoidable, this study focuses on extending the aging process by identifying optimal storage conditions that can be applied in the pulp industry.

Table 1. Dissolving pulp standard specification

Properties	Viscose fibres	Cellulose acetate	High ethers	viscosity
Intrinsic viscosity (ml/g)	450–500	650–900	> 1500	
Alkali resistance R18 (wt%)	94–97	98–99	~ 95	
Alkali resistance R10 (wt%)	89–92	94–98	~ 94	
Hemicellulose (xylan + man) (wt%)	3–5	1–2	4–6	
Acetone extractives (wt%)	0.2	0.05	0.1	
Lignin (kappa number)	< 0.3	< 0.2	< 0.5	
Total ash (wt%)	0.1	< 0.08	< 0.2	
Mn (ppm)	< 0.5	< 0.5	0.5	
Fe (ppm)	< 5	< 3	< 5	
Ca (ppm)	< 20	< 15	< 50	
Si (ppm)	< 20	< 10	< 15	
Brightness (%ISO)	89–93	< 92	> 85	
Density (g/cm ³)	~ 0.9	< 0.6	~ 0.8	

2 MATERIALS AND METHODS

2.1 Materials and Testing Instrumentations

These experiments were conducted using integrated industrial bleached hardwood dissolving pulp, with PHK cooking and ECF bleaching sequences of D0-EO-D1-D2. Samples of the pulp were collected after the final washing stage and diluted to a 10% consistency. The pulp's acidity, or pH, was adjusted using 1N HCl and 1N NaOH, both produced by the integrated pulp mill. The pulp quality was assessed before and after aging under specific conditions, with tests including viscosity measured by the Viscomat II and brightness evaluated using the Technidyne Brightimeter.

2.2 Aging Conditions and Testing Methods

The dissolving slush pulp sample is placed in a glass beaker and heated in a water bath for 144 hours to simulate natural aging, with varying pH levels (4, 6, and 8) and temperatures (50°C, 60°C, and 70°C). Quality analyses will be performed every 24 hours throughout the aging process. Viscosity will be measured according to SCAN-CM 15:99 and ISO 5351:2010 using the Viscomat II instrument. Brightness will be assessed based on TAPPI T 525 om-06 and TAPPI T-218 sp-02 standards. A hand sheet of the pulp will be prepared and conditioned at 23±1°C and 50±2% relative humidity before brightness readings is taken with the Technidyne Brightimeter. Table 2 provides an overview of the standard testing methods used to measure viscosity and brightness of the dissolving pulp before and after aging.

Table 2. The standard testing method used for analysis

Analysis	Testing Method
Viscosity	SCAN-CM 15:99
	ISO 5351:2010
Brightness	TAPPI T 525 om-06
	TAPPI T-218 sp-02

2.3 Cellulose Aging Kinetic Rate

The primary method for studying the aging process of pulp and paper involves applying the rate law of chemical reaction kinetics, as illustrated by Eq. (1). In this equation, C represents the concentration of reactants in a homogeneous chemical reaction system, n denotes the reaction order, t is the aging time, and k is the rate constant. For pulp and paper aging kinetics, C is substituted with properties such as fold endurance, tensile strength, tear index, brightness, and so on. When characterizing pulp and paper aging in terms of cellulose depolymerization, C can be replaced by the total number of unbroken glucose bonds in cellulose, with considerations for first-order, second-order reactions, and so forth. The rate of change in the degree of polymerization (DP) is described by Eq. (2) [13].

As shown in Eq. (2), k(t) represents the relative rate of bond breakage. Assuming a second-order reaction, integrating Eq. (2) results in Eq. (3). DP (t) denotes the number average degree of polymerization at time (t), while DP (0) represents the degree of polymerization of the pulp before aging. By plotting $\frac{1}{DP(t)} - \frac{1}{DP(0)}$ against time the reaction rate of cellulose bond breakage can be determined. In cellulose degradation, the degree of polymerization can be assessed by intrinsic viscosity values using the SCAN method. The average degree of polymerization (DP) is calculated from the intrinsic viscosity value as shown in Eq. (4), where η represents the intrinsic viscosity in cm^3/g [14].

$$\text{reaction rate} = \frac{dC}{dt} = -kC^n \quad (1)$$

$$\int_{DP=0}^{DP=DP} \frac{dDP}{DP^2} = - \int_{t=0}^{t=t} k dt \quad (2)$$

$$\frac{1}{DP(t)} - \frac{1}{DPn(0)} = kt \quad (3)$$

$$DP^{0.905} = 0.75 \eta \quad (4)$$

The specific rate of cellulose bond breaking in Eq. (2), denoted as k, is related to temperature according to the Arrhenius equation. While the Arrhenius equation is commonly used for single chemical reactions where both reactants and products are known, the cellulose aging process may involve multiple reactions. Consequently, the overall cellulose degradation rate constant, represented by Eq. (6), is the sum of the rate constants from each individual reaction and may not strictly follow the classical

Arrhenius equation. Where A_a and E_a represent the apparent frequency factor and activation energy of the multiple reaction system, respectively. The apparent activation energy is an average weighted by the rates of each reaction. This is why it is possible to obtain a linear Arrhenius plot for a multiple reaction system, even when the activation energies are very different [13].

$$k = \sum k_i = \sum A_i \exp\left(-\frac{E_i}{RT}\right) \neq A \exp\left(-\frac{E}{RT}\right) \quad (5)$$

$$k = A_a \exp\left(-\frac{E_a}{RT}\right) \quad (6)$$

3 RESULTS AND DISCUSSIONS

3.1 Cellulose Depolymerization Kinetic Rate

To examine the impact of aging on cellulose depolymerization, dissolving pulp samples were immersed in a water bath for a total of 144 hours. Viscosity reductions were measured at the 24th, 48th, 72nd, 96th, 120th, and 144th hours of aging. The degree of polymerization was calculated using Equation (4). Three different pH levels—4, 6, and 8—were applied to the pulp samples. The findings for all conditions are presented in Figure 3. Over the 144-hour aging period, viscosity decreased in relation to time, temperature, and pH. Both temperature and pH influenced viscosity reduction, with temperature having a more pronounced effect on accelerating the process compared to pH. The lowest viscosity observed after 144 hours of aging occurred at a temperature of 70°C and pH 4.

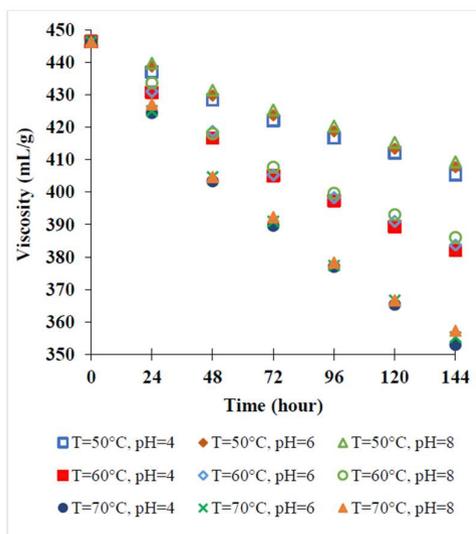


Fig. 3. Viscosity reduction in dissolving pulp aging over time.

Temperature speeds up the oxidation process, converting cellulosic hydroxyl groups into carbonyl structures (such as keto groups at C-2 or C-3, and aldehyde groups at C-6) or carboxyl groups (which can only form at C-6 and the reducing end) [12]. This oxidation modifies the cellulose chains, weakening their inter-chain bonds and leading to chain cleavage. As the cellulose chains shorten, both viscosity and degree of polymerization decrease. Acidity accelerates hydrolysis due to the presence of excess hydrogen ions (H^+), which protonate the C6 ring of HexA (hexuronic acid from hemicellulose), making it more vulnerable to nucleophilic attack by water molecules. This protonation weakens bonds, facilitating their cleavage during hydrolysis reactions [15]. In dissolving pulp, cellulose is present in greater quantities than hemicellulose. Consequently, during the aging process, temperature has a more significant impact on reducing viscosity compared to acidity.

To observe and compare the aging rates across the nine experimental aging variations, it is essential to determine the aging rate constants. The aging rate constant can be calculated by plotting $\frac{1}{DP(t)} - \frac{1}{DP(0)}$ against time, as outlined in Equation (3). Figures 4, 5, and 6 display these plots, showing how the aging rate constant, k was determined for the nine experimental conditions. The data from Figures 4 to 6 reveal that the aging rate constant increases with rising temperature. Specifically, at 70°C, the value of k is up to twice as high as it is at 50°C, indicating that pulp aging rate occurs up to twice at temperature 70°C compared to 50°C.

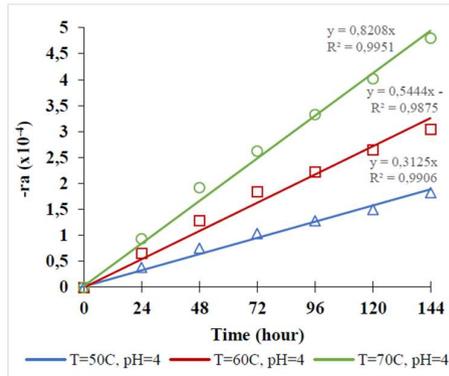


Fig. 4. Plot of dissolving pulp aging rate against time at pH 4

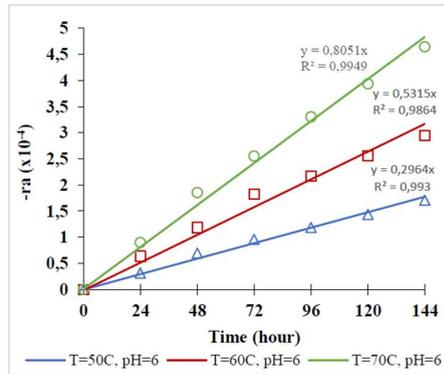


Fig. 5. Plot of dissolving pulp aging rate against time at pH 6

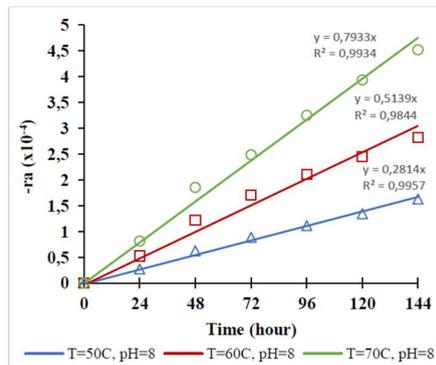


Fig. 6. Plot of dissolving pulp aging rate against time at pH 8

The aging rate constant, k , determined from the slopes in Figures 4 to 6, was then simulated in Python for comparison with the experimental data. Equation (1) was used with concentration (C) substituted by the degree of polymerization (DP), and least squares optimization was applied to obtain the optimized k value. Figures 7 to 9 show the comparison between the simulation and experimental data using optimized k value. The relationship between the simulation results and experimental data was sufficiently strong to conclude that the optimized k value can be used to predict the degree of polymerization (DP) of dissolving pulp over extended aging periods.

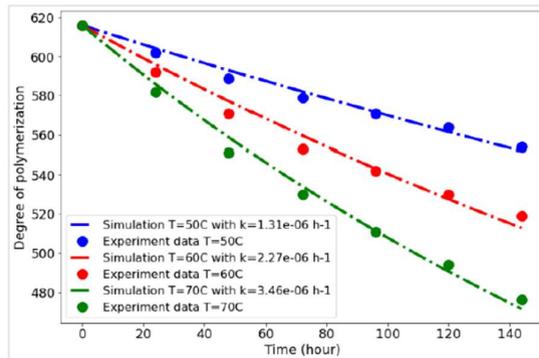


Fig. 7. Comparison of the simulation results for dissolving pulp aging, using the optimized k , with experimental data at pH 4.

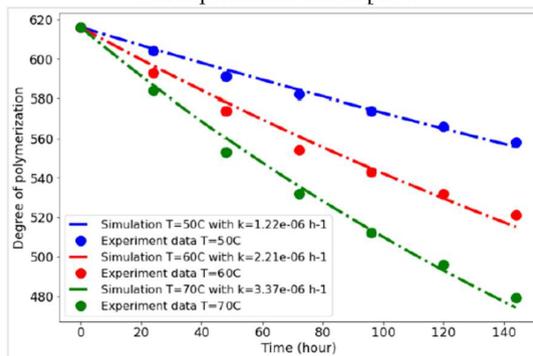


Fig. 8. Comparison of the simulation results for dissolving pulp aging, using the optimized k , with experimental data at pH 6.

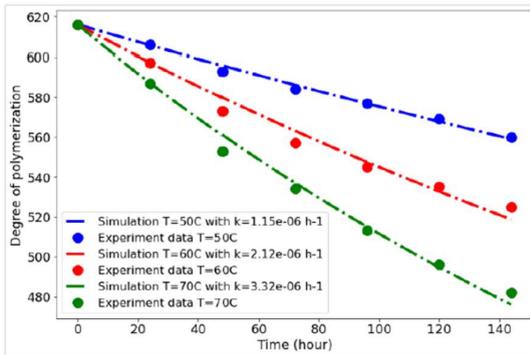


Fig. 9. Comparison of the simulation results for dissolving pulp aging, using the optimized k, with experimental data at pH 8

Table 3 compares the aging rate constants under nine different dissolving pulp aging conditions. The highest rate constant (k) was observed at 70°C and pH 4, while the lowest was at 50°C and pH 8. The maximum k value, $3.458 \times 10^{-6} \text{ h}^{-1}$ at 70°C and pH 4, is up to three times higher than the minimum k value at 50°C and pH 8, indicating that the aging rate is up to three times faster under these conditions. This comparison suggests that temperature has a more significant effect on the aging rate constant than pH. As temperature and acidity increase, the aging rate of the pulp accelerates, leading to higher k values.

Table 3. Dissolving pulp aging rate constant under different condition after six days aging

No.	Aging Condition		k, (hour ⁻¹) (x10 ⁻⁶)	Optimized k, (hour ⁻¹) (x10 ⁻⁶)
	T (°C)	pH		
1.	70	4	3.420	3.458
2.	70	6	3.355	3.375
3.	70	8	3.305	3.317
4.	60	4	2.268	2.269
5.	60	6	2.215	2.209
6.	60	8	2.141	2.116
7.	50	4	1.302	1.311
8.	50	6	1.235	1.222
9.	50	8	1.173	1.149

Figure 10 displays an Arrhenius plot of the rate constant, k, for cellulose depolymerization at pH 4, 6, and 8. It is evident that k is highly dependent on temperature and is linearly related to the inverse of temperature. This observation confirms the applicability of the Arrhenius equation to cellulose depolymerization during the aging of dissolving pulp. Regression analysis provided the apparent activation energy (Ea) for each pH condition, as detailed in Table 4. The activation energies for cellulose dissolving pulp degradation with natural aging in this study range

from 44 to 49 kJ mol⁻¹. The lowest activation energy occurs at pH 4, which is why the aging process is faster at this pH.

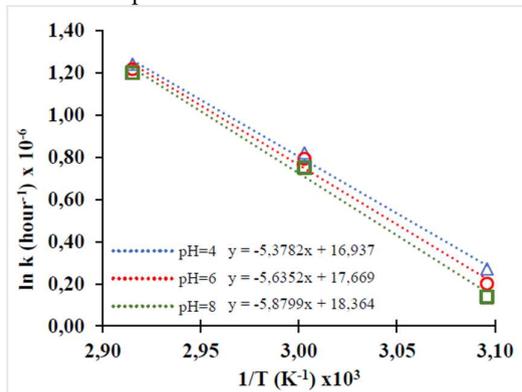


Fig.10. Arrhenius plot of rate constants of cellulose depolymerization.

Table 4. Frequency factors and activation energies for dissolving pulp aging

Sampel	a	b	Ea (kJ/ mol)
pH=4	16.937	5.378	44.717
pH=6	17.669	5.635	46.854
pH=8	18.364	5.880	48.888

3.2 Brightness Reversion

Brightness reversion is analyzed similarly to cellulose depolymerization. Figure 11 illustrates the effect of dissolving pulp aging on brightness reversal. Brightness diminishes over time, influenced by both temperature and acidity. Both factors significantly affect brightness reversion, with higher temperature and acidity accelerating the aging process. These findings are consistent with observations by previous researchers, who attribute the yellowing of celluloses to three stress factors: chemical (oxidative and hydrolytic) stress, photo-stress (light and irradiation), and thermal stress (temperature). Natural yellowing of pulp and paper, such as brightness reversion, occurs slowly at ambient temperature but accelerates with increasing temperature, acidity, and moisture content [16, 17]

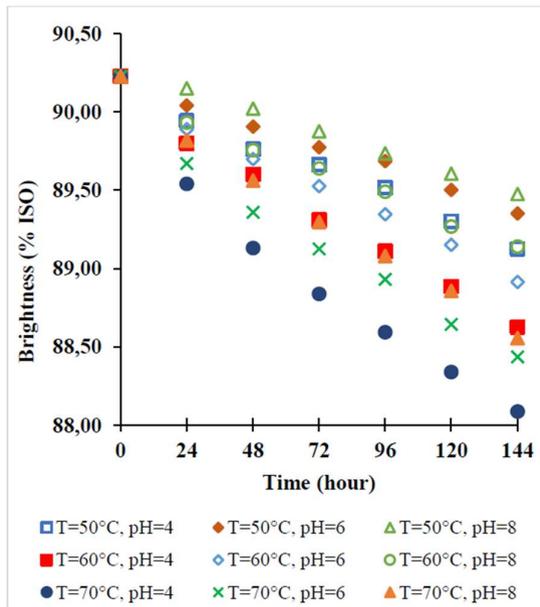


Fig. 11. Brightness reversion during the aging of dissolving pulp over time

Brightness reversion during pulp aging is caused by the formation of chromophore groups and the regeneration of chromophores destroyed in the bleaching process. During aging, oxidation converts cellulosic hydroxyl groups into carbonyl structures (keto groups at C-2 or C-3, aldehyde groups at C-6) or carboxyl structures (only possible at C-6 and at the reducing end) [12]. Carbonyl (C=O) and carboxyl (C=OOH) groups are recognized as initiators of yellowing; they are precursors to chromophores that form later during aging or yellowing [16]. Three main cellulose common chromophores identified after aging are: 2,5-dihydroxy- [1,4]-benzoquinones (DHBQ), 5,8-dihydroxy-[1,4]-naphthoquinones (DHNQ), and 2,5-dihydroxyacetophenones (DHAP) [18]. These chromophores significantly contribute to the overall color changes observed during aging.

A notable brightness reversion phenomenon is observed under pulp aging conditions at 70°C and pH 4. Under these conditions, brightness declines by up to 2% from its initial value within 6 days. To effectively reduce brightness reversion, it is advisable to lower the temperature to near ambient levels while increasing the pH to nearly neutral levels during storage. In contrast, at aging conditions of 50°C and pH 8, brightness losses are below 1% from the initial value. This suggests that brightness reversion can be mitigated by approximately three times compared to the more adverse condition described. Table 5 provide a detailed summary of brightness losses from the initial value across all dissolving pulp aging conditions, offering a clearer understanding.

Table 5. Dissolving pulp brightness losses under different conditions after six days aging

No.	Aging Condition		Brightness Losses (%)
	Temperature (°C)	pH	
1.	70	4	2.37
2.	70	6	1.98
3.	70	8	1.85
4.	60	4	1.77
5.	60	6	1.45
6.	60	8	1.20
7.	50	4	1.22
8.	50	6	0.97
9.	50	8	0.83

4 CONCLUSION

This study carefully examined how the quality of dissolving slush pulp declines under various aging conditions. The main findings are as follows:

1. The viscosity and brightness of dissolving pulp deteriorate noticeably over time during aging. The rate of this deterioration is significantly influenced by temperature and acidity levels.
2. There is a clear relationship between the brightness reversion of aging pulp and the length of cellulose chains (degree of polymerization). As these chains break down, brightness decreases, as demonstrated by the analysis of the aging rate constant (k).

To preserve the quality of dissolving slush pulp stored in tanks, it is essential to carefully control temperature and acidity levels. According to the research it is better to storage dissolving slush pulp at temperature 50°C with the pH at 8. The storage parameter adjusted to prolong the deterioration of cellulose chain as impact of natural agings.

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