

A Systematic Study on the Effect of the Xanthation Temperature on Viscose Quality [†]

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Abstract: The xanthation reaction is an exothermic reaction between alkali cellulose (AC) and carbon disulfide (CS₂) used to regenerate a viscose solution. The cooling system plays an important role during the reaction to yield more of the main product, cellulose xanthate (CX), instead of the by-product, sodium trithiocarbonate (TTC). Minimizing the yield of by-products during the reaction phase may lead to less by-product generation during the ripening process due to free caustic and excess CS₂ in the system. The reaction was performed in a batch reactor with an agitator (9.7 rpm) under vacuum conditions (350 mbar), and the temperature varied from 20 °C to 35 °C, as is applicable in industrial plants. Meanwhile, the CX and TTC were determined via UV spectroscopy. Since the temperature reaction will affect the period of the reaction, which impacts the productivity of industrial applications, the experiment was conducted with a temperature change during the reaction to obtain a good-quality product without impacting productivity. This work aimed to reach an optimum xanthation temperature under the same combination of hardwood and softwood dissolving pulp. The results indicated that the xanthation reaction has an advantage at lower temperatures compared to higher ones; however, having a lower temperature led to a longer reaction period. The TTC was shown to be 17.7% lower at lower temperatures than at higher temperatures, which means that the CX was at a higher percentage at lower temperatures. Interestingly, the combination of higher and lower temperatures gave good viscose quality, which may lead to less consumption of CS₂ and improve the environment due to less sulfur production during spinning.



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Keywords: xanthation; viscose; temperature; sodium trithiocarbonate; quality

1. Introduction

Viscose fiber is a man-made fiber formed by regenerating cellulose [1]. The high-purity cellulose, sourced from dissolving pulp (DP), is steeped into NaOH to increase the reactivity and remove impurities such as hemicellulose [2]. The penetration and bonding of Na⁺ ions to the cellulose molecules result in a material called alkali cellulose (AC), which can react with CS₂, the solvent of cellulose, to produce a viscose solution [3] in a reactor called a xanthator. CS₂ is a colorless, transparent fluid with a sweetish, aromatic odor. It boils at 40.4 °C and ignites spontaneously at about 115.6 °C but volatilizes at room temperature [4]. When injecting the CS₂ under vacuum pressure, the liquid-form CS₂ will transition into a vapor phase. In the xanthation process, AC reacts with vaporized CS₂ under alkaline conditions, followed by its dissolution in caustic soda and regeneration as viscose fiber under acidic conditions [5].

In the viscose industry, CS₂ is charged up to 30–35 wt.% of α-cellulose content in AC during xanthation, even though the requirement is 23.5 wt.% of α-cellulose content in AC for attaining the gamma number (the gamma number of viscose is an important characteristic, defined as the number of xanthate groups per 100 anhydrous glucose units (AHG) of the

cellulose [6]). At the same time, the α -cellulose content in AC will be determined via the gravimetric method. The distribution is as follows: 24% will be consumed in the basic reaction; 6.0–6.5% will be consumed in the forming of by-product compounds; and approximately 5% will remain in the free state [7]. The conversion of CS_2 into by-products, mainly TTC and Na_2S , will reduce the stability of the viscose dope, as indicated by the change in the CS_2 distribution in the cellulose molecule, the so-called ripening index (RI). The conversion of CS_2 into by-products will also lead to a viscosity change in the dope, generating viscose gel [8]. Reducing the by-product formation may reduce the CS_2 consumption as well and improve the quality of the viscose solution.

The availability of free caustic soda and hemicelluloses/low-molecular-weight cellulose encourages several CS_2 -consuming side reactions to take place. These variables may be minimized by obtaining control over the alkalizing process. Since the reaction between AC and CS_2 is a non-isothermal and nonadiabatic process [9,10], by-product formation is encouraged at higher temperatures, but dropping the xanthation temperature to achieve better CS_2 usage efficiency also extends the xanthation time, which affects production volume [11]. Moreover, during xanthation, CS_2 reacts principally with the C2 and C3 hydroxy substituents on the glucose rings in cellulose as these positions are kinetically favored over the C6 position due to the C6 position being thermodynamically stable. Any molecule will try to attain its thermodynamically stable state at C6 in CX [8,11]. Controlling the temperature will define the CS_2 molecule bonding in cellulose. The optimization of the temperature and xanthation time can be undertaken at a beneficial cost of production.

Kaller (1968) [10] increased the output by increasing the xanthation temperature without any modification of other processes. During the process, temperature variation occurred spontaneously with the variations in the final temperature, with each variant of the procedure not exceeding 1–2 °C. The temperature varied at 22, 27, 32, 37, and 42 °C. It was found that the increment in initial xanthation temperature from 22 to 42 °C will give a reduction in xanthation reaction time by almost 83.3%. This reflects that having a high reaction temperature will give more output of production. However, from the viscose quality point of view, the increment in initial xanthation temperature produces more by-product in viscose, i.e., from 8% to 10.2% with respect to α -cellulose content. By-product increment provides the most significant reflection of the lower esterification degree (ED) value and causes the ripening period to decrease, which may lead to gel formation [8].

There was another study on obtaining the maximum ED value on viscose via the variables of xanthation temperature and α -cellulose content [9]. Although there is no analysis of the by-product, the ED will reflect the by-product because of the CS_2 reaction with NaOH. The sharp decrease in the maximum degree of the esterification of cellulose xanthate at high xanthation temperatures (above 35 °C) and at a low α -cellulose content (less than 20%) was explained by the slower course of the process of carbon di-sulfide diffusion inside the cellulose molecule due to the alkali solution.

A recent study by Gondhalekar (2022) [8] has shown that the initial temperature was carried out at 25, 30, and 35 °C to study the rate of xanthate formation. The rate of xanthate formation is indicated in terms of % sulfur in xanthate form. The acceleration in the xanthation process happens when the temperature is raised; however, in the experiment, the by-product reaction also increases linearly with the increase in temperature.

The xanthation reaction between AC and CS_2 is a non-isothermal process [9]; hence, temperature plays a key role in deciding the direction of the reaction. This study emphasizes the impact of the cooling jacket temperature of the xanthator on the reaction. As the reaction temperature reduces, it may improve the quality of viscose by decreasing the TTC content in the viscose solution. However, the lower temperature led to a higher reaction time. This research aims to obtain optimum cooling temperatures to determine the most optimum reaction time and viscose quality. Based on the optimum temperature, the impact on the time injection of the cooling water into the system is considered to have optimum production cost.

2. Materials and Methods

The viscose dope used in this study was prepared at a standardized pilot plant using hardwood dissolving pulp (HWDP) from the Asia and softwood dissolving pulp (SWDP) from the USA. The composition of HWDP and SWDP was 96% HWDP + 4% SWDP, with a fixed CS₂ dosage of 32% of α -cellulose content in AC. Low SWDP composition, because the SWDP price is higher than HWDP (hence the low SWDP), is preferable on an industrial scale because of the operational cost. The properties of the dissolving pulp are shown in Table 1.

Table 1. Dissolving pulp properties [1].

Cooking Process	ASP	PHK
Raw material	Softwood	Hardwood
Brightness (%ISO)	90.2–90.8	90.6–98.1
α -cellulose (%)	92.4–94.8	94.2–96.3
Hemicelluloses (%)	3–5	2–3
Residual lignin (%)	<0.2	<0.2
Extractives (%) ^a	<0.3	<0.3
Ash (%)	0.1–0.2	0.1–0.2
Viscosity (cm ³ /g)	420–780	430–600
R ₁₈ content (%)	95.1–95.2	96.4–98.2
R ₁₀ content (%)	87.3–93.8	92.9–97.7
Fock Reactivity (%)	40–60	20–50
Chinese filterability (s)	n.d.	20–40
DP _w	4750	1400–2100
DP _n	450	460–650
PDI	10.6	3.0–4.5
DP < 100 (wt%)	0.5	2.0
DP > 2000 (wt%)	61.0	19.9–35.0
Degree of crystallinity (%)	n.d.	56

n.d. not defined; ^a Extractives in acetone.

The AC was fed into the reactor and then the reactor was set to vacuum conditions (200 mbar) and purged with nitrogen to 350 mbar. The reactor was isolated from the vacuum line, and a leakproof system was ensured by holding the vacuum condition for 5 min. Subsequently, CS₂ was added by using a flask for 10 min under vacuum conditions. The reactor was agitated at 9.7 rpm continuously during AC feeding till the reaction was finished. The reactor was also equipped with a cooling jacket. Here, a consistent range of coolant temperatures from 20 °C to 35 °C was upheld throughout the reaction using a water bath controller. After obtaining the optimum viscose quality, indicated by low TTC and high CX content with a reasonable reaction time, the cooling injection system was applied as well.

The finished point of the reaction was indicated by a tendentious pressure drop or pressure regain to the vacuum condition. Immediately after the reaction is finished, the 1 wt.% NaOH will be fed into the system to dilute the CX. Reactor pressure and temperature were recorded from the injection of the CS₂ step (time zero) until the pressure regained back to the vacuum. After dissolving the CX using NaOH, it is considered that the kinetics rate is significantly lower as the temperature of the NaOH was maintained at 4–5 °C.

The by-product (TTC) will be determined using UV spectroscopy HACH model DR-6000. A stock solution of 100 mL was prepared by diluting a gram viscose dope sample with 1% NaOH solution. A known amount of the sample (5 mL) was withdrawn from the stock solution and diluted to 250 mL with distilled water for UV spectrophotometric measurements. Absorbance values at 303, 332, and 363 nm were recorded within 10 min of dilution. Molar absorptivity values (ϵ) of the CX and TTC were obtained from the literature [12].

TTC will be the main by-product that will be calculated. The TTC concentration (CTTC, mole/L) was calculated with Equation (1). The correlation for absorption = 363 nm.

$$C_{TTC} = \frac{A_{363}}{\epsilon_{TTC}^{363}} = \frac{A_{363}}{2450}, \quad (1)$$

where A_{363} is the UV absorption and ϵ_{TTC}^{363} is the molar coefficient of absorption of TTC at 363 nm wavelength. The percentage TTC in a viscose solution can be estimated based on Equation (2), where 5.7×10^4 is a dilution factor [12].

$$\%_{TTC} = \frac{C_{TTC} \times 5.7 \times 10^4}{\text{weight of the sample in grams}} \quad (2)$$

Absorption at wavelengths of 303 nm and 332 nm corresponds to xanthate groups (C_{CX} , mole/L) in the viscose solution and can be calculated based on Equations (3).

$$C_{CX} = \frac{A_{303} - A_{332} \times 0.189}{15,900}, \quad (3)$$

where 15,900 is the coefficient of extinction for CX together with dioxanthogenide [12].

$$\%_{CX} = \frac{C_{CX} \times 3.2 \times 10^4}{\text{weight of the sample in grams}}, \quad (4)$$

where 3.2×10^4 is the dilution factor [12].

3. Results and Discussion

3.1. Alkali Cellulose Properties

The raw material was prepared with every experiment variable accounted for to avoid huge variances and to minimize the possibility of impacting the sulfurizing process, which is summarized in Table 2. The main parameter that will affect the quality of viscose given by product regeneration is the total alkali in AC, which shall not be of a high value (15%) [13,14]. The total NaOH and bonded NaOH were determined using distilled water, which was boiled to dissolve, combine, and neutralize the free compound with acid. This is shown in the reaction Equations (5) and (6) between AC and CS_2 . The excess of alkali does not give an advantage to the xanthation reactions as it will lead to more CS_2 consumption.

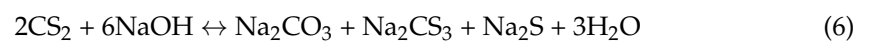


Table 2. Alkali cellulose laboratory properties before feeding to the xanthator.

S. N	Cooling Jacket Temperature	Pulp Weight	Consistency	White Spot	AC-TA	AC- α
		Gram	%	%	%	%
	Target	1700	4–5	/	14.5–15.5	33.5–34.5
1	20 °C	1700	4.71	7.94	16.31	35.45
2	25 °C	1700	4.68	2.89	16.18	35.94
3	30 °C	1700	4.49	4.59	16.12	36.81
4	35 °C	1700	4.78	5.86	16.33	35.92
5	30 °C to 25 °C at time of 20 min	1700	5.79	9.96	16.51	36.22
6	25 °C to 30 °C at time of 20 min	1700	4.86	4.64	15.8	36.2

In Table 1, we can see that all values exceeded the target requirement. The main reason is that the hydraulic pressing unit used is operated manually. Even when the pressure

increased from normal pressing (150 bar), the results were still on the higher side. However, the total alkali in the AC is slightly consistent at 16.5%, which shows that the impact of the high alkali content in AC can be neglected in this study.

Referring to the alkali content, the total bonded and free alkali were checked on every batch to obtain a detailed analysis before initiating the viscose dope. Table 3 shows the comparison between total alkali, bonded alkali, and free alkali in AC. With the same pressing unit, the bonded alkali shows a very consistent value. The higher bonded alkali in cellulose will lead to a better reaction with CS₂ given the product formation. However, the higher free alkali is a disadvantage in the production since the free alkali will bond to CS₂ during the gas-to-liquid transition of CS₂. Referring to Table 3, both samples 1 and 5 have less bonded alkali in AC and more free alkali in the liquid phase. This may lead to more by-product production than in other cases. Sample no. 4 has high free alkali content; however, the bonded alkali is also high, which may lead to high product and by-product generation during the reaction with CS₂.

Table 3. Laboratory results for bonded alkali in alkali cellulose before feeding to the xanthator.

S. N	Cooling Jacket Temperature	Total Alkali	Bonded Alkali	Free Alkali
		%	%	g/L
	Target	14.5–15.5	17–18	<8
1	20 °C	16.31	9.68	6.63
2	25 °C	16.18	9.64	6.54
3	30 °C	16.12	8.46	7.66
4	35 °C	16.33	9.10	7.23
5	30 °C to 25 °C at time of 20 min	16.51	8.72	7.79
6	25 °C to 30 °C at time of 20 min	15.8	8.34	7.45

Apart from the total alkali in AC, all data appear to be consistent across all parameters in Table 2. Sample no. 5 has high parameters, from the consistency of the slurry to the white spot, total alkali in cellulose, and high ball fall value on AC. Consistency will impact the pressing performance, which leads to high alkali content in AC. Meanwhile, a high white spot value may cause a high clogging value and filtration performance of the viscose due to cellulose unreacted with CS₂. However, all samples give consistent values that can compare the variables of the experiment.

3.2. Viscose Quality

In this study, the viscose quality was characterized by its main product (CX) and by-products (TTC). The xanthation product, which is viscose, was sampled at every stage of the process because it is not possible to check every minute during the xanthation reaction. Experiment data showed the same trend of product (CX) and by-product (TTC) production. The experiment data are given in Figures 1 and 2. During the initial reaction stage, the CX formation is much greater than the TTC formation. However, the CX content in the viscose is continuously decreasing at every stage, while TTC keeps generating during the process. In the reaction between AC and CS₂, three-OH groups are available on the C2, C3, and C6 carbon positions of the glucose chain, in which the C6 position is thermo-dynamically stable, whereas the C2 and C3 positions are the kinetically favorable positions. Hence, initial kinetics should be dominated by the CS₂ attack on C2 and C3 carbon positions, rather than C6. However, the CX content will keep decreasing due to the rearrangement of CS₂ from C2 and C3 of the glucose chain to C6. During the rearrangement, the released CS₂ will react with free NaOH, leading to the formation of a by-product [8,11].

The measurement of the results involved utilizing the percentages of each component (CX and TTC) as indicated by the instrument data as this study will concentrate on the trend itself.

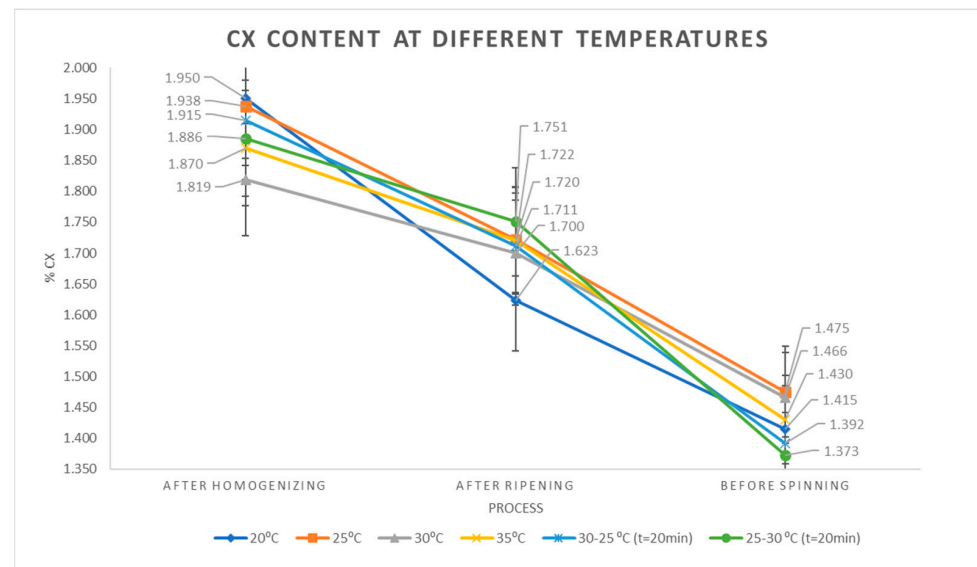


Figure 1. Product (CX) content at different xanthation temperatures and process stages based on laboratory results.

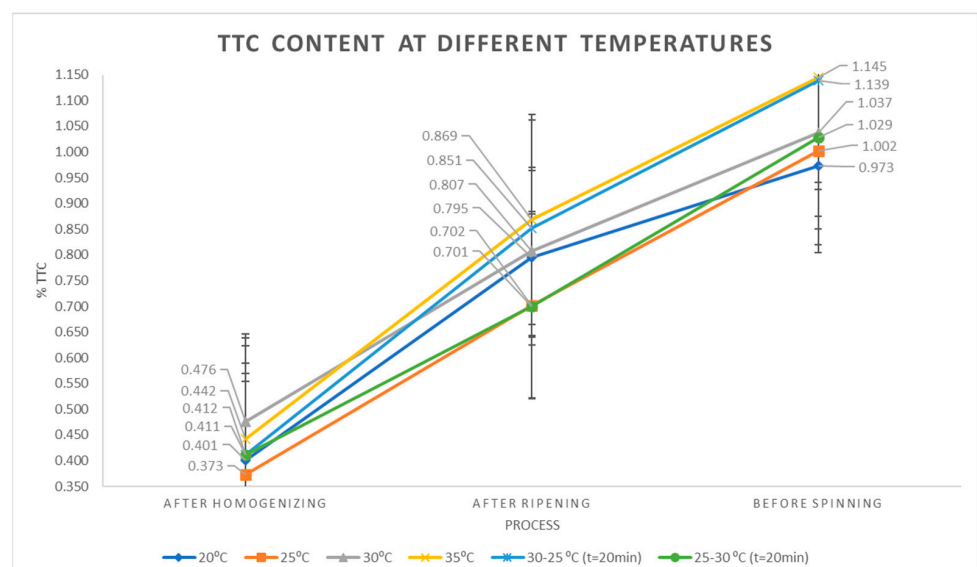


Figure 2. By-product (TTC) content at different xanthation temperatures and process stages based on laboratory results.

Based on Figure 1, CX formation was best achieved at 25 °C and 20 °C during the initial stage. However, the trend of CX content at 20 °C decreases with every process stage. At the end of the ripening period of viscose, viscose with 25 °C gives the best viscose, with high CX content for the spinning process. The reaction with 20 °C gives better results compared to the rest as the CX content was highest during initial process stage. Nevertheless, the decrease in CX content in the viscose is not satisfactory. The AC composition does exhibit much difference; especially with respect to total alkali content and free and bonded alkali in AC, it shall not impact the CX content. It is speculated that the degradation of CX content in the viscose is due to the regain time of the reaction. Experimental work by Kaller (1968) [10] has shown that every 5 °C increment of xanthation temperature shall yield a 15 min difference. However, the reaction was finished in 81 min, which may have caused the stability of the product and the CS₂ wastage during the reaction stoppage of this batch. Even though the xanthation with 20 °C gives better value than others, it is not able to be applied to the industrial scale due to the longer period of cycle batch production.

According to the xanthation period, the main product produced during 30 °C has a good result and a short xanthation period (45 min). Hence, both 25 °C and 30 °C are the best options for running a xanthation reaction while running a constant xanthation temperature.

During the rearrangement of C-bonding in AHU, the free CS₂ will react with free NaOH. Each stage of the process in viscose led to increased production of a by-product, as illustrated in Figure 2. The xanthation reaction with 25 °C xanthation temperature gives a lower value compared to 20 °C. However, the formation of TTC at 20 °C is less at the final stage compared to 25 °C. This shows that the initial temperature has a huge impact on the formation of by-products during the ripening period, which may slow down the TTC formation during the ripening period. Similar to CX formation, the 30 °C gives less TTC formation as well.

According to the individual cooling temperature result, both 25 °C and 30 °C are the best options at which to run the xanthation reaction. Therefore, both temperatures have been investigated further for cooling injection purposes. Option 1—the initial reaction was started by using a higher temperature (30 °C), and after 20 min, it was changed to a lower temperature (25 °C); Option 2—the initial reaction was started by using a lower temperature (25 °C), and after 20 min, it was changed into the higher temperature (30 °C). Given CX, Option 1 gives a slightly better result, which is 1.392% instead of 1.373%, as shown in Figure 1. Meanwhile, according to the TTC, Option 1 gives unfavorable results compared to Option 2. Option 1 gives 1.139% and Option 2 gives 1.029% (see Figure 2). Since the xanthation reaction is an exothermic reaction, temperature changes from high to low will cause the reaction to shift towards the product. This is consistent with Le Chatelier's principle, which predicts that a system at equilibrium will tend to shift in the endothermic direction if the temperature is raised. In this case, energy is absorbed as heat, and the temperature rise is opposed [15]. Lowering temperature shall be good in the reaction as it tends to consume CS₂ in the side reaction, which is conducive to regaining pressure in the reaction quickly. Nevertheless, in the xanthation reaction, it is not a good option as it will generate a side-product (TTC) instead of the main product. During the temperature change, the reactant, CS₂, changed from the gas phase to the liquid phase; hence, the excess CS₂ shall react with free NaOH [16], which was indicated by the pressure dropping during the reaction in Option 1, as shown in Figure 3. This may lead to more side-product production during the changes. Hence, the regain pressure is fast as the CS₂ finished fast to react with the free caustic.

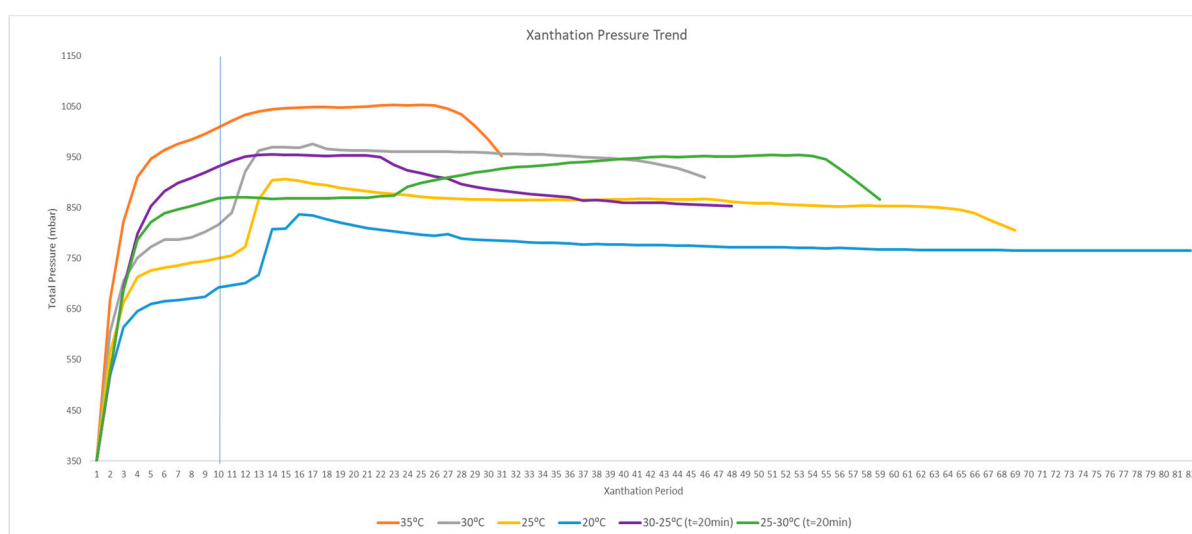


Figure 3. Total pressure as a function of the xanthation period at different xanthation temperatures based on laboratory results.

4. Conclusions

The impact of the xanthation temperature on the viscose quality has been systematically investigated. Xanthation is a reaction that involves a gas–liquid–solid system and an exothermic reaction. Xanthation cooling water temperature can be optimized at 25 °C and 30 °C. With the combination of both optimized temperatures, running with lower-to-higher temperatures is preferred. While running at a lower temperature is good for the system, it will impact the productivity of the plant due to the longer reaction period. We believe all running conditions can be implemented on an industrial scale.

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