

## ASSOCIATION OF NITROCELLULOSE WITH CUPRIC CHLORIDE IN SOLUTION\*

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### Summary

The cupric chloride (CC) effect on the viscosity of nitrocellulose (NC) in acetone solution was studied for various wood and cotton nitrocellulose samples with different nitrogen content and molecular weights. The addition of CC to NC solution, causes an increase of the viscosity as a function of time due to complex formation between NC and CC which is accomplished in two stages each with a specific rate constant. An empirical factor, the "Cupric Chloride Parameter" (CCP), is computed from the intrinsic viscosity of the polymer as well as from the kinetics parameters of complex formation and the weight fraction of CC relative to NC,  $X^*$ , determined at the saturation point. The CCP is characteristic of the NC sample and can be related to the cellulose source (wood or cotton) and nitrogen content. The value of  $X^{*1/2}$  can be used as a parameter to elucidate structural differences among NC samples.

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### 1.0 Introduction

It is well known that a variety of metallic and inorganic salts [1] and oxides [2] as well as free metals (especially Cu) can modify profoundly the properties of nitrocellulose (NC) in solution. The addition of inorganic salts to NC solutions causes a viscosity increase which is followed by gelation and eventually in many cases by syneresis [3].

Campbell and Johnson [4] found that the rate of gelling increased with the nitrogen content of NC indicating that residual hydroxyl groups were not directly involved in the gelation process. The gel formation seems to be due to the formation of a complex between the metallic salt and NC where cross-links are formed between the nitrate groups of adjacent NC chains. The rate of the association process followed an approximately first-order law and was accelerated by an increase in temperature.

Vodyakov [5] found that the cross-linker concentration is approximately proportional to the nitrogen content of NC. Bullock et al. [6] found that the increase in the viscosity of NC solution on the addition of lead  $\beta$ -resor-

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cylate is proportional to the molecular weight of NC. Jullander and Blom-Sallin [1] studied the cross-linking of NC solutions by means of titanium tetrachloride and found that cross-links are formed between unesterified hydroxyl groups of adjacent NC chains.

The object of this work is to relate the kinetics of complex formation between NC and cupric chloride (CC) to the structure, molecular weight and nitrogen content of NC as well to the cellulose source (wood or cotton). For this purpose, the CC effect on the viscosity of NC in acetone solution was studied for various wood and cotton NC samples of military and industrial grade, with different % N and molecular weights and obtained from different suppliers. The study was carried out at different shear rates and using different NC and CC concentrations.

## 2.0 Experimental

### 2.1 Samples

The characterized NC samples are listed in Table 1. The first eleven samples are typical of military applications whereas the last six samples are typical of commercial use. The reported values for the nitrogen content of the NC samples were provided by the suppliers.

The first three samples were obtained from Australia. Sample 1 was produced by the Explosives Factory at Maribyrnong from Maryvale pine pulp wood paper. Sample 2 was an experimental lot produced at Mulwala Explosives Factory from Australia Shoalhaven papered cotton linters. Sample 3 was a pyro wood NC made from Rayonier Q alpha cellulose board.

Samples 4–7 were obtained from EXPRO Chemical Products in Valleyfield, Quebec, Canada. Samples 4, 5 and 7 were produced from Rayonier Q-LD Woodpulp which meets requirements of Grade B of Spec. MIL-C-216-B. Sample 6 was produced from Buckeye Corp. Sheeted Cotton linters which meet requirements of Class III of Spec. MIL-C-206A. Samples 8 and 9 were prepared in DREV by blending samples 7 and 4 obtained from EXPRO respectively in a proportion 2 : 1 (sample 8) and 1 : 3 (sample 9). Samples 10 and 11 were obtained from the U.S.A. All the Pyro, HG and blend NC samples meet the U.S. Military Specifications (MIL-N-244A). Samples 12–17 are industrial NC samples obtained from Hercules Canada Ltd. in Toronto.

The solution properties of the NC samples are listed in Table 2. The intrinsic viscosity  $[\eta]$  values were determined at 25°C in acetone and the weight average molecular weight ( $M_w$ ) values obtained from Gel Permeation Chromatography (GPC) were determined using the NC calibration curve as described in Ref. [7]. The  $M_w$  values obtained by Low Angle Laser Light Scattering (LALLS) were determined by Dr. D. Gray through contracts [8,9]. The  $M_w$  values obtained from LALLS measurements are close to

TABLE 1

The characterized NC samples

Sample	Supplier	Cellulose source	% N	Description
1	Australia	wood	12.2	Displacement nitrated (1968), pine pulp
2		cotton	12.74	Mechanically nitrated (1974), papered cotton linters
3		wood	12.6	Mechanically nitrated (1974), $\alpha$ -cellulose board
4	EXPRO Chemical Products (Canada)	wood	12.6	Pyro (lot A-1-42), Grade "A"
5		wood	13.42	HG (from charge 2692), Grade "B"
6		cotton	13.14	Military Blend, Grade "C", Type I, from blend # C(1) 792-L
7		wood	13.43	HG (from charge 4712), Grade "B"
8		wood	13.15	Blend (2/3 sample 7 + 1/3 sample 4)
9		wood	12.80	Blend (1/4 sample 7 + 3/4 sample 4)
10	U.S.A.	cotton	12.6	Pyro (lot RAD PE 611-23), Grade "A", Type I
11		cotton	13.5	HG (lot RAD PE 611-24), Grade "B"
12	Hercules (Canada Ltd.)	wood	12.0 $\pm$ 0.2	RS 18-25 cps (lot 9055)
13		wood	12.0 $\pm$ 0.2	RS 5-6 s (lot 9057)
14		wood	12.0 $\pm$ 0.2	RS 1000-1500 s (lot 9060)
15		wood	12.3 $\pm$ 0.1	Extra-high viscosity (lot 965)
16		wood	11.5 $\pm$ 0.2	AS 3-6 s (lot 9062)
17		wood	11.0 $\pm$ 0.2	SS 40-60 s (lot 9054)

those obtained by GPC except for samples 15 and 17 which had a relatively high microgel content.

## 2.2 Instrument used

Viscosity measurements were determined at 20°C using a Haake ROTOVISKO Rotational Viscometer (Instrument No. 69-501). The viscometer was calibrated by using a standard liquid with the SV-I measuring system and at shear rates ranging from 3.3 to 529 s<sup>-1</sup>. The viscosity ( $\eta$ ), shear rate ( $G$ ) and shear stress ( $P$ ) were obtained respectively from the following relations

$$\eta = USA \quad (\text{cp})$$

$$G = 529/U \quad (\text{s}^{-1})$$

$$P = G\eta/1000 \text{ (Pa)}$$

where  $U$  = speed factor,  $S$  = scale reading and  $A$  = calibration constant.

### 2.3 Solutions studied

The cupric chloride ( $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ) used was obtained from Fisher Scientific Company. The CC in a 1.2 wt.% acetone solution was added to the NC also in acetone solution. The CC weight fraction relative to NC( $X$ ) varied between 0.2 to 18 wt.%. The NC concentration ( $C$ ) in the mixture was between 1.2 and 4.2 wt.% depending on the  $X$  value and the sample studied.  $X$  is related to  $C$  by the expression  $X = m/(C+m)$  where  $m$  is the CC concentration in wt.%. The value of  $C$  was chosen in such a way to allow viscosity determination with a good precision and for at least 5–7 h before gel formation because a low value for  $C$  will yield viscosity measurements lacking precision whereas a high  $C$  value will produce a rapid gelation.

TABLE 2

Solution properties of the NC samples

Sample	$[\eta]$ acetone, dl/g	$M_w 10^{-5}$	
		GPC	LALLS <sup>a</sup>
1	3.20	2.1	1.76
2	3.00	2.5	2.11
3	3.50	2.2	1.86
4	4.50	2.7	2.34
5	3.40	2.7	
6	3.25	2.0	
7	2.83	1.9	
8	3.40	2.2	
9	3.88	2.5	
10	3.40	2.0	
11	4.0	2.1	
12	0.40	0.22	0.28
13	1.41	0.77	0.70
14	4.00	2.75	3.12
15	10.60	3.80	8.46
16	1.30	0.73	
17	1.60	1.10	1.81

<sup>a</sup>Data from Refs. [8] and [9].

## 3.0 Results and discussion

### 3.1 Cupric chloride effect on the solution viscosity

The addition of CC to the NC solution causes an increase of the viscosity as a function of time as is shown in Fig. 1 where  $\log \eta/\eta_0$  is plotted against the time ( $t$ ) for sample 2.  $\eta$  and  $\eta_0$  are respectively the viscosity

at time  $t$  and the viscosity at  $t = 0$ . This increase of the solution viscosity is probably due to cross-linking reaction or chelate formation between CC and the nitrate groups or the residual hydroxyl groups or both. The increase of viscosity that occurs leads to the formation of complexes which is followed after a certain time by gelation. This phenomenon will be discussed in section 3.3.

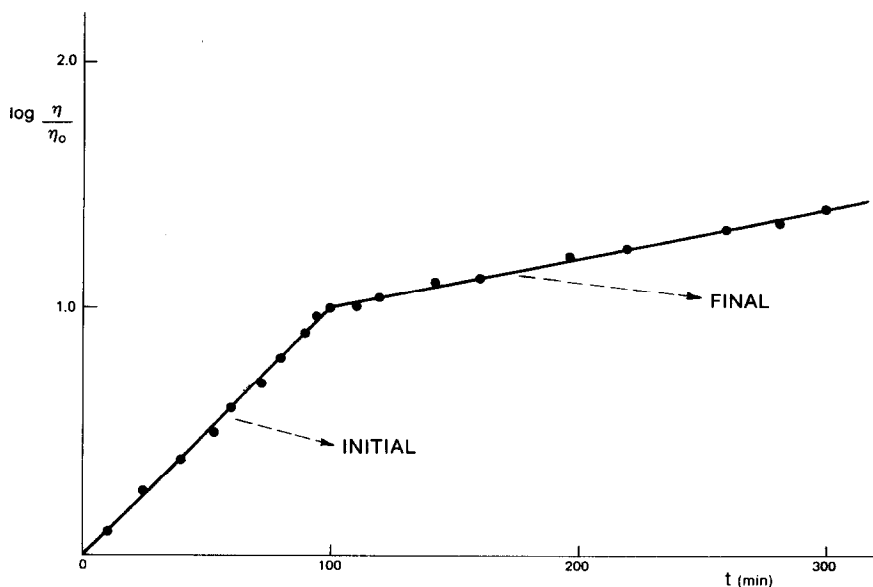


Fig. 1. Variation of viscosity with time for sample 2 at  $X = 6.0\%$ ,  $C = 2.6\%$ ,  $G = 59 \text{ s}^{-1}$ .

All the NC samples studied yielded complexes on the addition of CC except sample 12 which had the lowest  $M_w$  (22,000) indicating that the complexes begin to form only when the molecular weight exceeds a certain critical value.

As shown in Fig. 1, the graph of  $\log \eta/\eta_0$  vs.  $t$  yields two straight lines, each with a distinct slope. All the NC samples studied presented this common behaviour. If we assume that the rate of complex formation is proportional to the increase of viscosity with time (represented by the slope), then the complex formation between CC and NC is probably accomplished in 2 stages, each with a specific reaction velocity and rate constant. The first stage has a rate constant proportional to the initial slope ( $k_i$ ) and the second stage has a rate constant proportional to the final slope ( $k_f$ )

$$k_i = \frac{d(\log \eta/\eta_0)}{dt} \quad \text{initial}$$

$$k_f = \frac{d(\log \eta/\eta_0)}{dt} \quad \text{final}$$

The  $(k_i/k_f)$  value represents actually the ratio of the initial stage rate constant to the final stage rate constant. In most cases  $k_i$  is higher than  $k_f$  and the ratio  $(k_i/k_f)$  depends on the NC sample studied and the chosen  $X$  value as will be shown in the next sections.

### 3.2 Effect of $C$ on the rate of complex formation

The values of  $k_i$  and  $k_f$  as well as the ratio  $(k_i/k_f)$  obtained for sample 8 at  $X = 7.7\%$  and  $G = 59 \text{ s}^{-1}$  are reported in Table 3 for 4 different  $C$  values. The values of  $k_i$  and  $k_f$  increase with  $C$  but the ratio  $(k_i/k_f)$  for a given  $X$  value is constant ( $5.4 \pm 5\%$ ) and independent of the NC concentration in the mixture. For this reason, the ratio  $(k_i/k_f)$  will be used particularly for comparison purposes among different NC samples.

TABLE 3

Effect of  $C$  on the rate of complex formation for sample 8 at  $X = 7.7\%$  and  $G = 59 \text{ s}^{-1}$

$C$ (%)	$k_i 10^3$ ( $\text{min}^{-1}$ )	$k_f 10^3$ ( $\text{min}^{-1}$ )	$(k_i/k_f)$
1.80	4.9	0.9	5.3
2.42	6.1	1.2	5.1
2.80	7.7	1.4	5.3
3.16	11.3	2.0	5.7

### 3.3 The gel point

In the course of complex formation between NC and CC, the gel point occurs at a defined stage when the mixture transforms from a viscous solution to a gel and the viscosity measurements become no longer possible. The time at which this phenomenon happens, is the gelling time ( $t_{\text{gel}}$ ) which is reported in Table 4 for 4 NC samples and at different  $C$  and  $X$  values. We notice that  $t_{\text{gel}}$  varies with the proportions of reactants as well with the  $[\eta]$  value according to the empirical relation

$$[\eta]^{1/2} X^{1/2} C^2 t_{\text{gel}} = 1.15 \times 10^5 \pm 15\% \text{ min ml}^{1/2} \text{ g}^{-1/2}$$

This relation was computed from the experimental results and holds for the 4 NC samples as shown in Table 4.

As  $[\eta]$  is proportional to the NC molecular weight through the Mark-Houwink relation, then we can conclude that gel formation occurs more readily the higher the molecular weight and the NC concentration which is in agreement with [10]. It is possible of course to avoid gelation by varying the NC and CC proportions in the mixture.

### 3.4 Effect of $G$ on the rate of complex formation

The process of complex formation between NC and CC for all the samples studied followed a non-Newtonian behaviour where the viscosity de-

creases with increasing shear rates as shown in Fig. 2 where  $\log \eta_G$  is plotted against  $G$  for sample 5 at  $t = 435$  min;  $\eta_G$  is the viscosity at  $G$  shear rate. It was found empirically and in the case of all the NC samples, that the graph of  $\log \eta_G$  vs.  $\log 1/(G+1)$ , which is also drawn in Fig. 2 for sample 5, is a straight line whose intercept yields the value of  $\eta_{G_0}$  (the viscosity at zero shear rate).

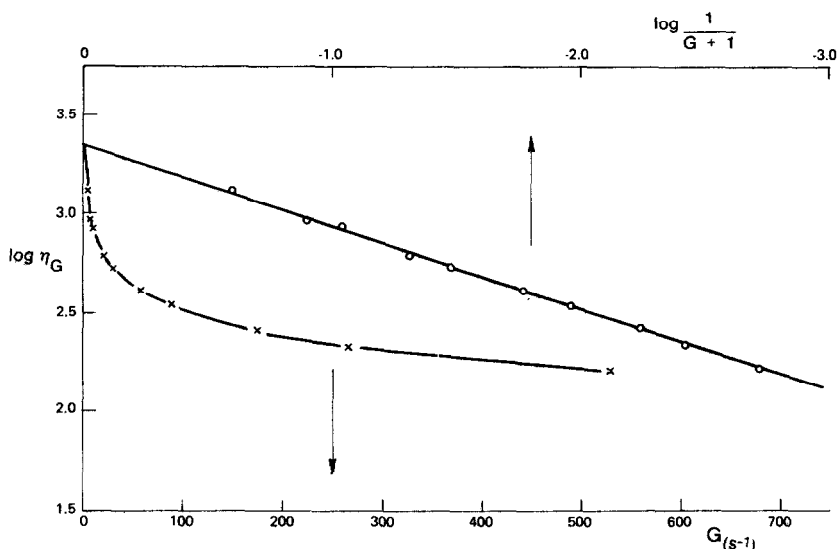


Fig. 2. Variation of viscosity with shear rate for sample 5 ( $t = 435$  min,  $X = 1.7\%$ ,  $C = 2.3\%$ ).

TABLE 4

Effect of  $C$  and  $X$  on the gelling time for different NC samples

Sample	$[\eta]$ (dl/g)	$C$ (%)	$X$ (%)	$t_{\text{gel}}$ (min)	$(X^{1/2} [\eta]^{1/2} C^2 t_{\text{gel}}) 10^{-5}$ (min ml <sup>1/2</sup> g <sup>-1/2</sup> )
15	10.60	2.46	1.5	460	1.1
		2.46	3.0	300	1.0
		2.46	5.9	210	1.0
8	3.40	3.54	4.1	255	1.2
		3.03	5.9	320	1.3
		2.42	8.0	430	1.3
17	1.60	3.43	6.2	325	1.2
		3.43	11.3	265	1.3
		3.05	13.0	310	1.3
16	1.30	4.21	8.2	190	1.1
		3.45	11.7	280	1.3
		2.63	18.2	360	1.2

The process of complex formation between NC and CC has a pseudo-plastic flow behaviour as shown in Fig. 3 where the graph of  $P$  vs.  $G$  is drawn for sample 5 at  $t = 375$  min.

The values of  $k_i$  and  $k_f$  as well as the ratio  $(k_i/k_f)$  for sample 15 for a  $X$  value of 5.8% and at 4 different shear rates are reported in Table 5. From the results listed in Table 5, we notice that the values of  $k_i$  and  $k_f$  decrease with increasing shear rates whereas the ratio  $(k_i/k_f)$  is constant ( $18.4 \pm 5\%$ ) and independent of  $G$  for a given  $X$  value. For comparison purposes, all the reported values of  $(k_i/k_f)$  were determined at  $G = 59 \text{ s}^{-1}$ .

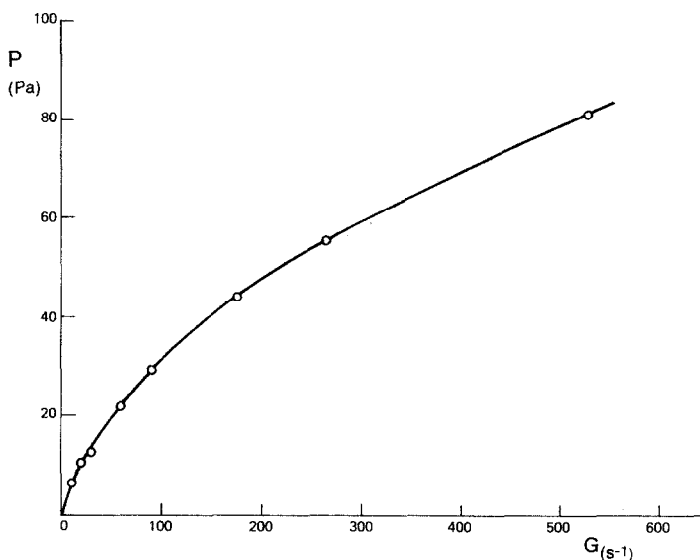


Fig. 3. Variation of  $P$  with  $G$  for sample 5 ( $t = 375$  min,  $X = 1.7\%$ ,  $C = 2.3\%$ ).

TABLE 5

Effect of  $G$  on the rate of complex formation for sample 15 at  $X = 5.8\%$  and  $C = 1.23\%$

$G$ ( $\text{s}^{-1}$ )	$k_i 10^3$ ( $\text{min}^{-1}$ )	$k_f 10^3$ ( $\text{min}^{-1}$ )	$(k_i/k_f)$
3.3	11.2	0.58	19.3
10	7.9	0.45	17.5
59	6.1	0.33	18.5
265	4.0	0.22	18.0

### 3.5 Effect of $X$ on the rate of complex formation

The variation of the ratio  $(k_i/k_f)$  with  $X$  is shown in Fig. 4 for sample 11 at  $G = 59 \text{ s}^{-1}$ . From the graph we notice that the ratio  $(k_i/k_f)$  increases with  $X$  until it reaches a maximum value  $(k_i/k_f)^*$  at a certain critical value



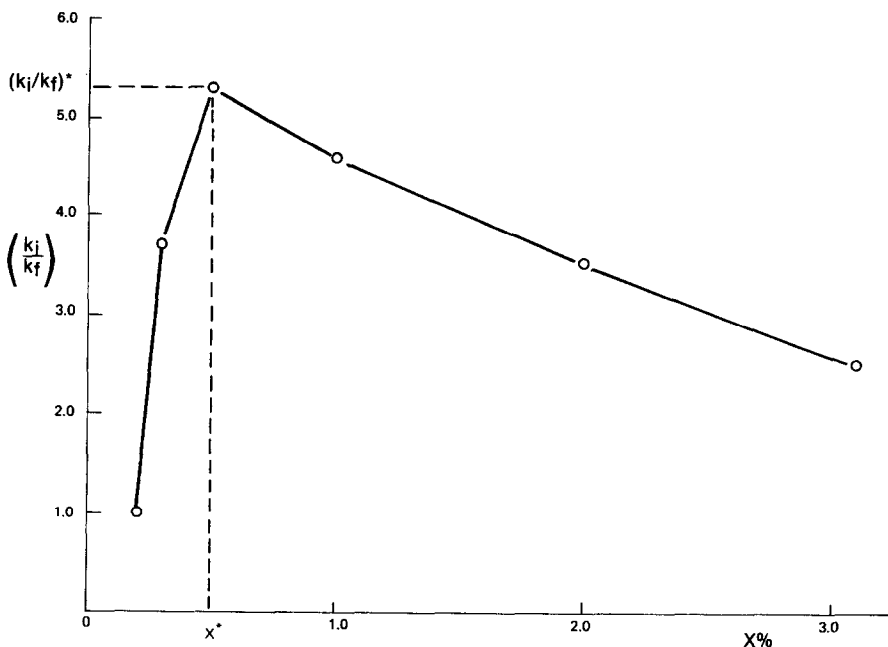


Fig. 4. Variation of  $(k_i/k_f)$  with  $X$  for sample 11.

$X^*$  (the saturation point) and then starts to decrease. All the NC samples studied presented this common behaviour. These values of  $X^*$  and  $(k_i/k_f)^*$  obtained at the saturation point, are characteristic of the NC sample studied and are related to the polymer structure, the molecular weight, nitrogen content and cellulose origin as will be shown in the next sections.

The values of  $X^*$  and  $(k_i/k_f)^*$  obtained for all the NC samples studied are listed in Table 6. The asterisk sign designates the results obtained at the saturation point.

### 3.6 Structure effect

In this paper, the word "structure" refers to the distribution of the nitrate and residual hydroxyl groups along the polymer chains. In order to study the structure effect on the rate of complex formation, we compared the data of NC samples produced from the same cellulose source and with similar nitrogen content and  $[\eta]$  values. In this study, we chose the  $[\eta]$  rather than the molecular weight ( $M$ ) because the  $K$  and  $a$  parameters in the Mark-Houwink relation  $[\eta] = KM^a$ , are also related to the structure [11,12].

The results obtained at the saturation point for 4 wood NC samples are reported in Table 7. We notice that  $X^*$  decreases with  $(k_i/k_f)^*$  from a value of 9.0 (sample 1) to a value of 3.0 (sample 4). According to these experimental results, the value of  $(k_i/k_f)^*$  is directly proportional to  $X^{*1/2}$

TABLE 6

The results obtained at the saturation point for the NC samples

Sample	$X^*$ (%)	$(k_i/k_f)^*$
1	9.0	7.8
2	4.5	7.4
3	6.2	7.8
4	3.0	5.7
5	1.4	8.8
6	1.9	7.0
7	2.1	7.1
8	7.7	5.7
9	2.1	5.0
10	7.6	5.6
11	0.5	5.3
12	—	—
13	11.8	2.9
14	5.0	6.8
15	5.8	18.5
16	8.4	3.4
17	11.3	4.8

TABLE 7

Effect of structure on the rate of complex formation

Sample	$X^*$ (%)	$(k_i/k_f)^*$	$(k_i/k_f)^*/X^{*1/2}$
1	9.0	7.8	2.6
3	6.2	7.8	3.1
14	5.0	6.8	3.0
4	3.0	5.7	3.3

as indicated in Table 7 where the ratio  $(k_i/k_f)^*/X^{*1/2}$  has a constant value ( $3.0 \pm 10\%$ ) for all the 4 NC samples. Although all these samples are wood NC with approximately the same % N ( $12.3 \pm 0.3$ ) and  $[\eta]$  values ( $3.8 \pm 0.6$ ), they present different structures as shown by their different  $X^*$  values. As CC probably forms complexes with the nitrate or unesterified hydroxyl groups or both, then the distribution of these functional groups around the glucose units and along the cellulose chains varies from one NC sample to the other. The value of  $X^{*1/2}$  can then be used as a structural parameter to differentiate between NC samples with similar molecular weights and degree of nitration. We hope that the  $X^{*1/2}$  value could be used in a simple routine test to distinguish between NC samples which meet all the military specifications but nevertheless yield gun propellants with different ballistic properties.

### 3.7 Molecular weight effect

In order to study the molecular weight effect on the rate of complex formation, we compared the data of NC samples with the same cellulose source and nitrogen content but with different  $[\eta]$  values. The results obtained for 4 wood NC samples are reported in Table 8 along with the values of  $(k_j/k_f)^*/X^{*1/2}$  which take into account structural differences among the samples.

TABLE 8

Molecular weight effect on the rate of complex formation

Sample	$[\eta]$ (dl/g)	$(k_j/k_f)^*/X^{*1/2}$	$(k_j/k_f)^*/[\eta]X^{*1/2}$ (g/dl)
15	10.60	7.7	0.73
14	4.00	3.0	0.75
1	3.20	2.6	0.81
13	1.41	0.84	0.60

From the results listed in Table 8, we notice that  $[\eta]$  increases with the ratio  $(k_j/k_f)^*/X^{*1/2}$  whilst the ratio  $(k_j/k_f)^*/[\eta]X^{*1/2}$  is nearly constant ( $0.70 \pm 15\%$  g/dl) for all the 4 NC samples. This ratio  $(k_j/k_f)^*/[\eta]X^{*1/2}$  denoted the "Cupric Chloride Parameter" (CCP) is an empirical factor characteristic of the NC sample as will be shown in the next section.

### 3.8 Effect of nitrogen content and cellulose origin

The CCP values computed from the experimental data for all the NC samples studied are reported in Table 9. We notice that the CCP value is constant ( $0.77 \pm 20\%$  g/dl) for all the wood and cotton NC samples with % N below 12.7. Although these samples were obtained from different suppliers and had a nitrogen content between 11% and 12.6%, their CCP value was independent of the cellulose origin and the degree of nitration. However, for cotton NC samples, the CCP value increases with the degree of nitration as shown in Table 9 where the CCP value varies from 0.60 g/dl (% N = 12.6) to 1.85 g/dl (% N = 13.5). This change in the NC properties which occurs at approximately 12.7% N, was reported by other authors who investigated this phenomenon using other techniques such as viscosimetry [13], IR spectroscopy [14] and X-ray diffraction [15]. The variation in the CCP value observed for cotton NC at 12.7% N which corresponds to a degree of substitution of 2.5, results probably from the change in NC properties occurring at the transition from dinitrate to trinitrate. No change in the CCP value was observed in the case of wood NC samples with % N below 13.4. Both wood and cotton NC of the High Grade type presented a common behaviour as shown in Table 9 where the CCP values reported for sample 5 (2.21 g/dl) and sample 7 (1.73 g/dl) are close to the one obtained for sample 11 (1.85 g/dl).

TABLE 9

Effect of % N and cellulose source on the CCP

Sample	% N	Cellulose source	CCP (g/dl)	
17	11.0	Wood	0.89	
16	11.5		0.90	
13	12.0		0.60	
14	12.0		0.75	
1	12.2		0.81	
15	12.3		0.73	
3	12.6		0.89	
4	12.6		0.73	
9	12.8		0.89	
8	13.15		0.60	
5	13.42		2.21	
7	13.43		1.73	
10	12.6		Cotton	0.60
2	12.74			1.16
6	13.14	1.60		
11	13.5	1.85		

#### 4.0 Conclusions

The process of complex formation between NC and CC in acetone solution is accomplished in 2 distinct stages (initial and final), each with a specific rate constant. The ratio of the initial stage rate constant to the final stage rate constant is independent of  $G$  and  $C$  but depends rather on the NC sample and the chosen  $X$  value.

The ratio  $(k_i/k_f)^*$  and the corresponding  $X^*$  value obtained at the saturation (critical) point, yield valuable information on some important properties of the characterized NC sample such as the structure and the cellulose source (wood or cotton).

An empirical factor (CCP) which is specific and characteristic of the NC sample is computed from the critical values of  $X^*$  and  $(k_i/k_f)^*$  obtained at the saturation point as well as from the  $[\eta]$  value of the polymer. Wood and cotton NC have a common CCP value for samples of the HG type (% N = 13.5). The CCP value can be useful to differentiate between wood and cotton NC samples mainly in the range  $12.6 < \% N < 13.4$ . In that range, the CCP value is constant for wood NC whilst it increases with the degree of nitration in the case of cotton NC.

The  $X^{*1/2}$  value is a structural parameter that can be used in a simple routine test to distinguish between NC samples with similar molecular weights and nitrogen content.

From the results obtained in this study, we cannot conclude whether the nitrate or the residual hydroxyl groups of NC were directly involved

in the process of complex formation with CC. To clarify this point, other techniques such as nuclear magnetic resonance should be investigated.

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