

**Research/Technical Note**

Kinetic Study of the Esterification Between Citric Acid Molecules and *Dombeya Mollis*' Leaf Cellulose Titrated by Spectrophotometer UV-Visible Procedure

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Abstract: Cellulose is the most abundant natural organic polymer on earth and could be valorized and used in different applications. It's mainly used to produce paper but also to produce biodegradable cellophane and biofuels. Thus, the extraction of cellulose from green plants, where it's the most important structural component, was interesting. This manuscript treated the extraction of cellulose from the *Dombeya Mollis leaf* by esterification-reaction with citric acid molecules. This method is usually used to extract, to identify, to characterize and to quantify the active molecules of alive organisms but in this case the advantage to use the esterification-reaction with citric acid molecules is that the synthesized solution contains straightaway reticulated cellulose. Indeed, the refractive index value of the *Dombeya Mollis leaf's* cellulose-citric acid esterified solution was 1.336 in the vicinity of the refractive index values of citric acid-solid, citric acid in solution and nanocellulose which were respectively 1.493, 1.345 and 1.470. In addition, A cellulose quantification procedure using spectrophotometer UV-visible was performed and established in this manuscript and permitted to quantify the quantities of cellulose extracted from the plants to the solution by esterification-reaction with citric acid molecules. In consequence, a kinetic study was done under this esterification-reaction between citric acid molecules and *Dombeya Mollis leaf's* cellulose and permitted to evaluate the speed constant with the partial orders of citric acid, cellulose and proton-H⁺ which play the role of catalyst-inductive during this esterification-reaction. At the end, a reactional mechanism which could govern this esterification-reaction between the *Dombeya Mollis leaf's* cellulose and the citric acid molecules was proposed.

Keywords: Citric Acid, Cellulose, Esterification, *Dombeya Mollis*, Leaf, Spectrophotometer UV-Visible, Kinetic, Reactional Mechanism

1. Introduction

The first part of this manuscript treated the description of the experimental conditions used during this

esterification-reaction between the *Dombeya Mollis leaf's* and the citric acid molecules which was carried out at 144°C using a reflux assembly [1-6]. It described also the principal subject of this manuscript such as the description of the titration

procedure to quantify cellulose in the reactional medium and the titration of the citric acid molecules. The second part is dedicated to the cellulose titration procedure application into the analysis and exploitation of the experimentations results to determine the kinetic parameters of this reaction-esterification between the citric acid and the *Dombeya Mollis* leaf's cellulose. And finally, the third part consisted to propose and to elaborate a reactional mechanism which could govern this esterification-reaction. Materials and chemicals used during experimentations were KERN precision scale, chronometer, glass spatula, iron spatula, refractometer, magnetic stirrer, magnetic bar, flask-250ml, heating mantle-250ml, condenser, beaker-100ml, beaker-250ml, beaker-400ml, graduated burette, pipette, citric acid, *Dombeya mollis* leaf's leaf distilled water, NaOH-0.05N, helianthine, saturated iodine solution 3.34[g/l], spectrophotometer UV-visible.

2. Experimental Conditions and Quantifications Procedure Used During This Kinetic Study

The bibliography said that citric acid was used to reticulate the cellulose in order to synthesize reticulated cellulose which physical-chemical properties were improved to resist on

tension, to resist on water or formic acid dissolution and to have a thermic stability [7]. In this manuscript, direct esterification-reactions [1-6] between citric acid molecules and cellulose from *Dombeya Mollis* leaf's was done to product a reticulated citric acid-cellulose solution. The citric acid conversion and the cellulose quantities in the reactional medium was followed-up with time reaction.

2.1. Experimental Conditions for the Kinetic Study of the Esterification-Reaction Between the Citric Acid Molecules and Celluloses from *Dombeya Mollis* Leaf

Esterification-reactions with citric acid was usually used to extract, to identify, to characterize and then to quantify the active organic molecules of alive organisms [4-7]. This esterification-reaction was done in a reflux assembly composed with a balloon-250ml putted in a balloon-heater and a condenser at the top as described in the bibliographies [1-6]. The following table 1 show the experimental conditions of this esterification-reaction where a certain sample quantities of the reactional medium was taken at an exact instants and titrated in order to quantify not only the extracted cellulose and its derivatives like reticulated-cellulose transferred in the reactional medium but also the left-over of the citric acid molecules in the reactional medium.

Table 1. Experimental conditions of the *Dombeya mollis* leaf esterification-reaction with citric acid.

<i>Dombeya mollis</i> leaf's esterification-reaction with citric acid							
Weight of the <i>Dombeya mollis</i> leaf [g]	9,0589						
<i>Dombeya mollis</i> leaf Nature	Cut out into small cylinder or cubes or square forms the <i>Dombeya mollis</i> leaf						
Water content of the <i>Dombeya mollis</i> leaf's [%]	68,33						
<i>Dombeya mollis</i> leaf color	green ± brown transparent						
Distillated water volume [ml]	200						
Citric acid weight [g]	14						
Calculated-pH	2,02						
Temperature (°C)	144						
Total reaction duration [h]	1h						
Sample remove reaction time [s]	60	180	300	600	900	1800	3600
Extract solution color after esterification	Dark green						
Total extract Volume [ml]	126						
Extract refractice index	1336						
Left-over leafs color	Dark green						
Left-over leafs weight [g]	78						
Characteristics of the left-over leafs	Grains-speck and cylindrical Easily friable						
Taste	Bitter and acide						

2.2. Quantification Procedure of Citric Acid and Cellulose Molecules in the Reactional Medium

2.2.1. Quantification Procedure of Citric Acid in the Reactional Medium

The citric acid molecules left-over in the reactional medium was evaluated by a titration procedure described in the bibliography [1, 2] where 1ml-sample was removed from the reactional medium every considered reaction time using a pipette and transferred into a beaker-250ml. Dilute this sample into 15ml of distillated water then add three drops of the color indicator helianthine. The titrated solution color turns into orange-red color. Start the titration with the NaOH-0.05N

putted into the burette and stop the titration when the solution color turns into yellow-orange color. Record the equivalent NaOH-0.055N volume and deduced the quantity of citric acid left-over in the reactional medium at this instant.

2.2.2. Quantification Procedure of Cellulose in the Reactional Medium

The literature said that under concentrated sulfuric acid during a certain instant, the cellulose turns into amyloid and the presence of iodine in the solution could turn its color into blue (Case of the industrial synthesis of amyloid from the unsticked papers plunged during few seconds in sulfuric acid-80% [8]). In this publication, a procedure for the quantification of cellulose into a solution or a product was

inspired. The chemical products used during this cellulose quantification were concentrated 80% sulfuric acid and iodine saturated solution. The concentrated 80% sulfuric acid was prepared from concentrated 90% sulfuric acid solution. And, the iodine concentrated solution was prepared by dissolving 0.0167g of iodine into 5ml of distilled water, agitated on the magnetic stirrer during at least 7mn until having a yellow orange solution with few iodine grains undissolved. This saturated iodine solution have a weight concentration equals to 3.34[g/l].

This cellulose quantification procedure starts with taking a sample equivalent to 1ml solution even if the material to be titrated is solid or liquid and putting it into a test tube. Then, add 12ml of the concentrated 80% sulfuric acid which, in presence of cellulose, induced the smoked formation. Finally, add 0.2ml of the iodine solution 3.34[g/l] which turned the solution color from yellow-orange transparent until light-brown or dark-brown to black according to the sample cellulose content as shown the following Figure 1.

The samples in the test tube were leaved and covered during at least 7hours before their spectrophotometer UV-visible analysis at the wave length 610nm which correspond to the detection of amyloids-derivatives from cellulose mentioned by the bibliography [9, 10]. In fact, the cellulose concentration

of the sample is deduced by this amyloids-derivatives concentration given by this spectrophotometer UV-visible analysis.



Figure 1. Colorations of the sample after concentrated 80% sulfuric acid treatment with adding 0.2ml of iodine 3.34[g/l].

Indeed, be $X \left[\frac{\mu\text{g}}{\text{ml}} \right]$ the concentration of the amyloids detected by spectrophotometer UV-visible; let's supposed that it's a amylopectin, thus it's molar is equivalent to 100000 and with a factor 55.68 is deduced the total moles of amyloids-amylopectin such as

$$\text{Moles}_{\text{amylopectine}} = 55.68 \times \left(\frac{X \times 10^{-6} \times \text{Volume of the solution to be titrated}}{100\,000} \right) \quad (1)$$

Thus, seeing that the amylopectin is a ramification of 10^{+5} à 10^{+6} molecules of D-glucose/cellulose so the total quantities of cellulose is

$$\text{Moles}_{\text{cellulose}} = 10^{+6} \times \text{Moles}_{\text{amylopectin}} \quad (2)$$

And, the molar concentration of cellulose in the total sample is

$$[\text{cellulose}] \left[\frac{\mu\text{g}}{\text{ml}} \right] = \frac{\text{Moles}_{\text{cellulose}} \times 10^{+6} \times \text{Molar mass}_{\text{cellulose}} (162.1406)}{\text{Volume solution to be titrated}} \quad (3)$$

In consequence,

$$[\text{cellulose}] \left[\frac{\mu\text{g}}{\text{ml}} \right] = 9027,9886 \times X \left[\frac{\mu\text{g}}{\text{ml}} \right] \quad (4)$$

3. Kinetic Study of the Esterification-Reaction Between the Citric Acid and Cellulose

3.1. Presentation of the Experimental Results

Noticed that a determination of the *Dombeya mollis* leaf's cellulose content was carried out and it is equal to 28.29%. And, the results of this esterification-reaction between the citric acid molecules and the *Dombeya mollis* leaf's cellulose is shown in the following Table 2 and Figure 2. Noticed that the values of the amylopectin concentration at 60s, 600s and 900s were negative and couldn't be presented in this table but would be confirmed and eventually completed. However,

their concentrations of cellulose were determined by modelisation.

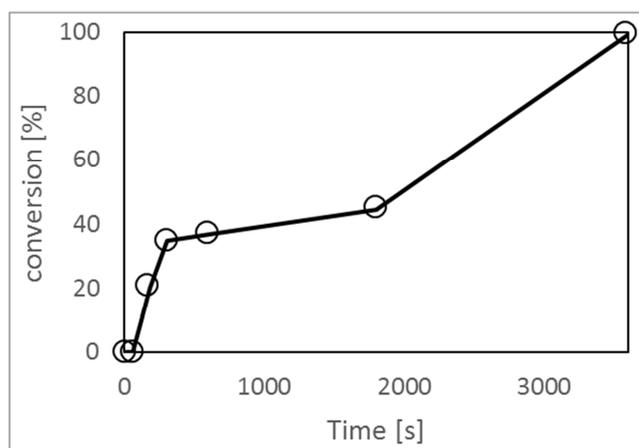
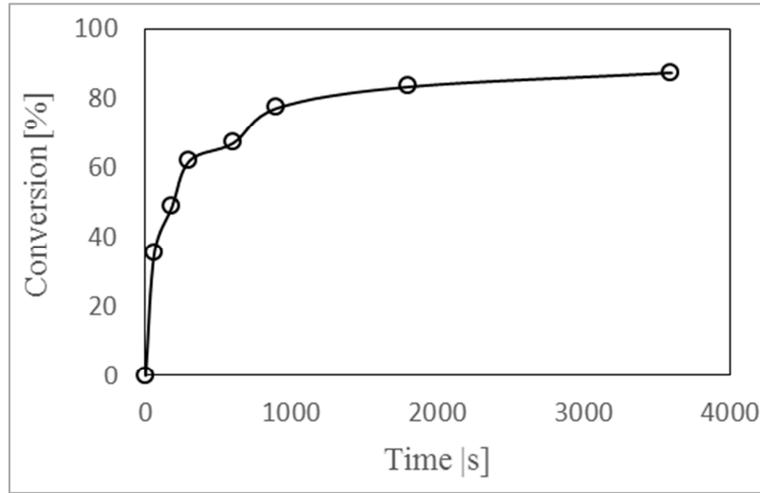


Figure 2. Evolution of the cellulose conversion with the reaction time.

Table 2. Esterification-reaction between the citric acid molecules and the *Dombeya mollis* leaf's cellulose results.

Reaction time [s]	60	180	300	600	1800	3600
amylopectin concentration [$\mu\text{g/ml}$]	6,7E-3	2,88E-3	4,94E-2	5,22E-2	6,31E-2	14,1E-2
cellulose concentration [$\mu\text{g/ml}$]	60,488	260,0061*	445,98264	471,261	569,666081	1272,9464
Cellulose concentration [mol/L]	2.48E-06	1,60E-3	2.75E-03	2.91E-03	3.51E-03	7.85E-03
Cellulose [g]	4,68E-05	1,81E-04	8,92E-02	9,33E-02	1,14E-01	2,55E-01
conversion cellulose - χ [%]	0.03	20.29	34.80	36.78	44.46	99.34
conversion citric acid (%)	35.47	48.785	62.097	67.23	83.61	87.65
[Ac] ⁻ [mol/L]	2.35E-01	1.87E-01	1.38E-01	1.19E-01	5.97E-02	4.50E-02
[H ⁺][mol/L]	1.01E-03	9.73E-04	9.53E-04	9.31E-04	9.03E-04	8.91E-04

*Estimated value

**Figure 3.** Evolution of the citric acid conversion with the reaction time.

3.2. Kinetics Parameters Evaluation During This Esterification-Reaction

The speed of the esterification-reaction between the *Dombeya mollis* leaf's cellulose and the citric acid molecules seeing that the protons - H⁺ were catalysts-inductive during this reaction:

$$v(\text{réaction\%cellulose}) = \frac{-1}{(V \times v_i)} \times \frac{d[C_{\text{cellulose}}]}{dt} = k_{\text{see}} \times [\text{acide citrique}]^\alpha \times [\text{cellulose}]^\beta \times [H^+]^\omega \quad (5)$$

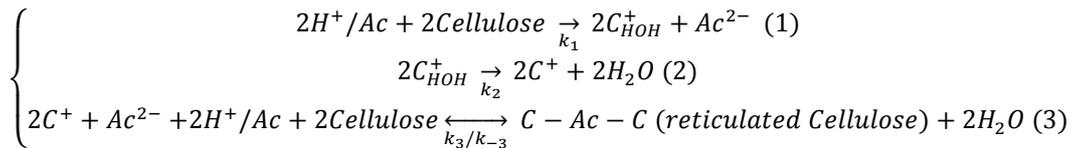
Applying the base 10 logarithmic function on the previous equation-5 using the results of the four initial points in the table 2, the values of the four kinetic parameters on the equation-5 was determined and shown in the following table 3.

Table 3. Kinetics parameters of the esterification-reaction between the *Dombeya mollis* leaf's' cellulose and the citric acid molecules.

α	β	ω	k_{see}
-0.1351	+1.23E-2	+2.2	2.84×10^{662}

4. Reactional Mechanism Proposed During This Citric Acid-Cellulose

Esterification The reactional mechanism proposed to govern this esterification-reaction between the *Dombeya mollis* leaf's' cellulose and the citric acid molecules is



Thus, the speed of the reticulated cellulose is

$$v_{\text{formation reticulated cellulose}} = k_3 [C^+]^2 [Ac^{2-}]^1 [H^+]^2 [Cellulose]^2 - k_{-3} [H_2O]^2 [C - Ac - C] \quad (6)$$

Applying the quasi-stationary-state theory on the reactional intermediaries C_{HOH}^+ and C^+ , the following expressions could be deduced

$$\frac{d[C^+]}{dt} = k_2[C_{HOH}^+]^2 - k_3[C^+]^2[Ac^{2-}][H^+]^2[Cellulose]^2 + k_{-3}[C - Ac - C][H_2O]^2 = 0 \quad (7)$$

$$\frac{d[C_{HOH}^+]}{dt} = k_1[H^+]^2[Cellulose]^2 - k_2[C_{HOH}^+]^2 = 0 \Leftrightarrow [C_{HOH}^+]^2 = \frac{k_1}{k_2}[H^+]^2[Cellulose]^2 \quad (8)$$

$$\Rightarrow [C^+]^2 = \frac{k_1}{k_3[Ac^{2-}]} + \frac{k_{-3}[C-Ac-C][H_2O]^2}{k_3[Ac^{2-}][H^+]^2[Cellulose]^2} \quad (9)$$

Using this expression on the equation-6 (speed expression) thus,

$$v_{\text{formation reticulated cellulose}} = k_3[C^+]^2[Ac^{2-}][H^+]^2[Cellulose]^2 - k_{-3}[H_2O]^2[C - Ac - C]$$

$$v_{\text{formation reticulated cellulose}} = \left(k_3 \left(\frac{k_1}{k_3[Ac^{2-}]} + \frac{k_{-3}[C-Ac-C][H_2O]^2}{k_3[Ac^{2-}][H^+]^2[Cellulose]^2} \right) [Ac^{2-}][H^+]^2[Cellulose]^2 \right) - k_{-3}[H_2O]^2[C - Ac - C] \quad (10)$$

After development and simplification, the expression of the speed is

$$v_{\text{formation reticulated cellulose}} = k_1[H^+]^2[Cellulose]^2 \quad (11)$$

After drawing the evolution of the reticulated cellulose speed formation with the expression $[H^+]^2[Cellulose]^2$, the following figure 4 is deduced by choosing adequate points and the value of $k_1 = 26643$.

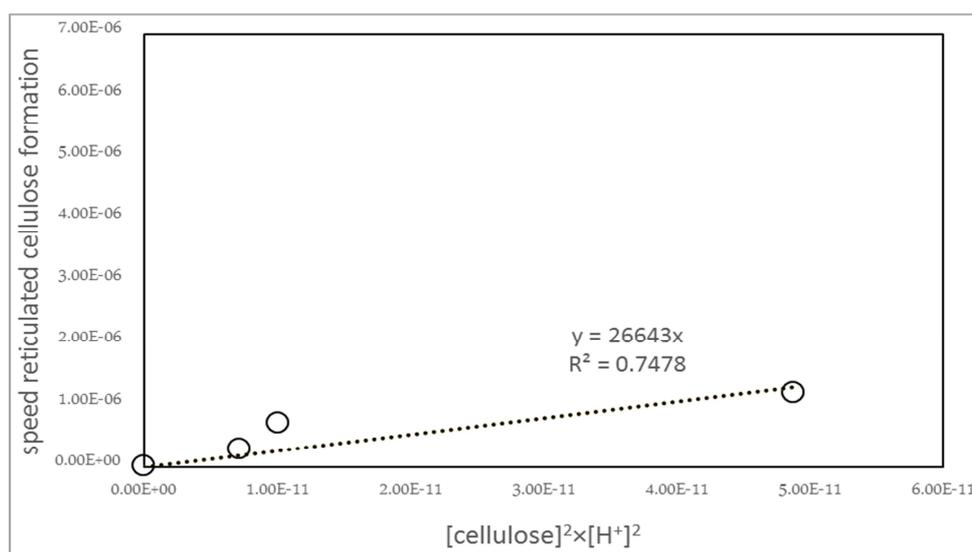


Figure 4. Evolution of the reticulated cellulose speed formation with the expression $[H^+]^2[Cellulose]^2$.

5. Conclusion

The results of the experimentations carried out in this manuscript showed that the citric acid molecules are efficiency to extract the cellulose of the earth like the *Dombeya mollis*' leaf. Indeed, the refractive index of the citric acid ester of the *Dombeya Molli leaf's* molecules solution was 1.336 such as a part was soluble dichloromethane. This value was in the vicinity of the refractive index values of citric acid-solid, citric acid in solution and nanocellulose which were respectively 1.493, 1.345 and 1.470 [11, 12] and indicated the presence of citric acid reticulated cellulose – C-Ac-C- [§4 - equation3] which is soluble in dichloromethane like citric acid molecules. In addition, the cellulose quantification procedure based on its transformation into amyloids and derivatives using the concentrated 80% sulfuric acid added with saturated iodine solution 3.34[g/l] produced right and valid results from which the determination of the kinetics constants of the esterification-reaction between the

Dombeya mollis leaf and the citric acid molecules at 144°C was done and it was confirmed the role of the proton-H⁺ as catalyst-inductive said on the literatures [13-17]. At the end, the expression of the reticulated cellulose speed-formation according to the expression $[H^+]^2 \times [Cellulose]^2$ as a straight line confirmed the proposed reactional mechanism which take into account the possible rehydration of the reticulated citric acid cellulose. It was also confirmed from this reactional mechanism that the proton-H⁺ partial order is two (2), a correct value to a catalyst-inductive. It was also noticed that the cellulose partial order and the speed constants k determined from their evaluation by the experimental results and from the proposed reactional mechanism were respectively multiple.

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