

Research/Technical Note

Trisodium Citrate Synthesis at Ambient Temperature Using Sodium Hydroxide Relatively in Excess and Citric Acid with Ethanol-90 as Solvent

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To cite this article:

Nambinina Richard Randriana, Avotra Marson Randrianomenjanahary, Andry Tahina Rabearitsara. Trisodium Citrate Synthesis at Ambient Temperature Using Sodium Hydroxide Relatively in Excess and Citric Acid with Ethanol-90 as Solvent. *American Journal of Applied Chemistry*. Vol. 9, No. 3, 2021, pp. 83-89. doi: 10.11648/j.ajac.20210903.14

Received: June 6, 2021; **Accepted:** June 18, 2021; **Published:** June 25, 2021

Abstract: Trisodium citrate $C_6H_5O_7Na_3$ is one of the most widely used synthetic chemicals in the laboratory as raw materials to synthesize some metals-citricacid-oxides and also to control the acidity of certain substances and/or solutions; thus it is rare to find it in stock at suppliers. That was why synthesizing this product in laboratory came suddenly in the head and ended up being realized within certain experimental conditions such as: the synthesis was carried out at room temperature, under atmospheric pressure, the initial concentration of sodium hydroxide 4.38 twice higher was relatively in excess against citric acid and using ethanol-90° as solvent. It consisted of mixing citric acid with sodium hydroxide 99% purity, and ethanol 90% purity which served as a solvent. In fact, the two raw materials, citric acid and sodium hydroxide are all soluble in ethanol while the desired product trisodium citrate isn't insoluble in this solvent-ethanol. The calculated initial pH of the raw materials solution was 15.21 and the solvent-ethanol quantities was evaluated so that eventual water molecules and eventual sodium ethoxide formed during the synthesis were soluble in it. Thus, all synthesized trisodium citrate crystals insoluble in ethanol were located at the bottom and separated. To quantify all these synthesized trisodium citrate- $C_6H_5O_7Na_3$ crystals, a titration procedure using hydrochloric acid HCl-0.1N was established. Application of this established titration procedure allowed to follow the reaction with time and consequently to evaluate the speed constant of this reaction synthesis of trisodium citrate- $C_6H_5O_7Na_3$ crystals under these previous experimental conditions which was equal to $1.56E-2 [L^2 \times mol^{-2} \times s^{-1}]$. Also, the initial speed formation of synthesized sodium ethoxide was equal to $0.0027 [mol \times l^{-1} \times s^{-1}]$ and the citric acid conversion only after 30 minutes of reaction was 92.56%. The evaluation of this synthesized trisodium citrate crystals porosity was done by immersion in an isopropyl alcohol giving a value in the order of 48,67% and density equal to $1.79 [g/cm^3]$.

Keywords: Trisodium Citrate, Citric Acid, Ethanol, Isopropyl Alcohol, Sodium Hydroxide, Hydrochloric Acid, Titration

1. Introduction

First, this laboratory synthesis of trisodium citrate crystals $C_6H_5O_7Na_3$ was carried out using sodium hydroxide (99% purity) relatively in excess 4.38 twice higher over citric acid with ethanol (90% purity) as a solvent. Citric acid and sodium

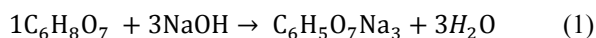
hydroxide all being soluble in the volume of ethanol used. A precision balance KERN, beakers-250[ml], a test tube 50[ml], a magnetic stirrer-Fischer Scientific, a stirring rod, a water bath Büchi B-480, a pH-meter Isolab, a burette-50[ml], a desiccator, a pipette of 10[ml], citric acid, sodium hydroxide-99%, ethanol-90% and helianthine color indicator were materials and chemicals used in the synthesis. Then, the

titration of synthesized trisodium citrate solution was carried out with hydrochloric acid HCl-0.1N using a burette-50[ml] and a helianthine color indicator according to a method operative described below. This titration procedure allowed the evaluation of the molar conversion of citric acid. Finally, the total crystallization of the concentrated trisodium citrate being completed, the porosity of this crystal was determined by immersing 0.0174 [g] of dehydrated trisodium citrate crystals in 15 [ml] of absolute alcohol using a crystallizer covered with a bell jar and a beaker-250[ml] to avoid humid air condensation during 150[mn].

2. Experimental Conditions of Trisodium Citrate- $C_6H_5O_7Na_3$ Synthesis

The raw materials used to synthesize this trisodium citrate ($C_6H_5O_7Na_3$) were citric acid and sodium hydroxide 99% (NaOH) with ethanol 90° as solvent seeing that all used citric acid and sodium hydroxide during each synthesis experimentations were soluble in the used ethanol [1, 2]. In addition, the sodium hydroxide was relatively in excess compared to the citric acid. As shown in the following table 1, the molar ratio of sodium hydroxide-NaOH divided by citric acid- $C_6H_8O_7$ was equal to 4.38. Indeed, the stoichiometric of this synthesis in the following figure 1 showed that the sodium

hydroxide was experimentally and theoretically in excess if the previous ratio was equal to $(4 \times (3:1))$, but on this actual experimental conditions this ratio was just adjusted to $(1.46 \times (3:1))$ which economized this NaOH-raw material.



Noticed also that, on this actual experimental conditions, not only all used NaOH-raw material were completely soluble in $C_2H_5OH-90^\circ$ but also all used citric acid $C_6H_8O_7$ -raw material were completely soluble in $C_2H_5OH-90^\circ$ during synthesis (Table 1) seeing that the sodium hydroxide solubility in ethanol was 139[g/l] [2-6] and citric acid solubility in ethanol was 62[g]/100[g] [1], so not only the raw materials collisions certainly increased efficiently but also the rest of raw materials were also completely soluble in the used ethanol according to this experimental conditions. The approximate calculated pH of the solution initially were 15.21. But, the recorded pH of the solution after the first mixing were 8.5 to increase at a maximum value 11.17 seeing that it was composed with two strong bases ethanol and sodium hydroxide which were largely in excess compared to citric acid moles with a ratio equal to 73.22 [7, 8]. Thus, noticed that this 8.5 of the solution only after the first mixing (Table 1) was already in the vicinity of a dehydrated Trisodium citrate pH=8.4 [9].

Table 1. Experimental conditions of trisodium citrate- $C_6H_5O_7Na_3$ synthesis using sodium hydroxide – citric acid and ethanol-90° as solvent.

Raw materials	Ethanol- $C_2H_5OH-90^\circ$ (solvent)	Sodium hydroxide NaOH-99%	Citric acid
Initial volume [ml]	21	-	-
Weight [g]	-	0.9259	1.0054
Raw materials quantities [moles]	0.3239	0.0230	5.23E-3
Initial raw materials concentrations [mol.l ⁻¹]	15.4226	1.0906	2.49E-1
Raw materials molar ratio divided by sodium hydroxide	14.08	1.00	0.2283
Reaction temperature	Ambient temperature		
Reaction durations [s]	60 – 90 – 900 – 1,800		
Maximum raw materials solubility in ethanol-90° [g]	-	2.6271	9.2508
Calculated - pH	15.21		
Recorded pH after first mixing	8.5		
Maximum recorded pH after first mixing	11.17		

3. Experimental Procedure of Trisodium Citrate- $C_6H_5O_7Na_3$ Synthesis

First of all, weight the used sodium hydroxide-0.9259[g] and the used citric acid-1.0054[g] with a precision balance, put them into a beaker-250[ml] making sure that half of the beaker was occupied by NaOH and the other half was occupied by citric acid without mixing (figure 1). Then, placed the stirring rod. Then, measure the volume-21[ml] of ethanol used with test tube-50[ml]. Put the beaker-250[ml] on a magnetic stirrer, begin to stir slowly and insert all at once all ethanol with increasing slowly the magnetic stirrer speed and agitating the beaker efficiently in such a way all sodium hydroxide and citric acid was dissolved with formation of homogeneous white-transparent solution. Few seconds later began the formation of trisodium citrate crystals which have the particularity of being insoluble in ethanol [10] and settle at

the bottom of the beaker [11] following the stirring rod. According to the enthalpies of formation of the compounds on the reaction (1) given by bibliographies [12-15], the standard enthalpy of this reaction was equal to +1970.906[KJ/mol]. These results indicated and confirmed that not only this reaction is slightly endothermic [14], but also the present experimental conditions and procedure with the solvent-ethanol 90° put together were efficient to produce the trisodium citrate. When the reaction time was achieved, remove the beaker-250[ml] from the magnetic stirrer to put it over a water bath less than 288.15°C to freeze and to stop the reaction during 15 minutes. Then, a heterogeneous solution composed with ethanol slightly white-transparent and trisodium citrate crystals was obtained. Transfer only the liquid-ethanol slightly white-transparent to another second beaker-250[ml] and let it decant. Remove the liquid-ethanol slightly white-transparent of this last second beaker without removing the rest of trisodium citrate crystals; then rinse and

transvase it into the first beaker-250[ml] with maximally 15[ml] of ethanol-90°. Let decant the very heterogeneous solution for few minutes and remove ethanol using water bath 333.15[°K] for few minutes until the smell of alcohol was less felt. Once synthesized the amount of trisodium citrate was defined by titration with hydrochloric acid 0.1N [16-17]. Noticed that all the synthesis was at ambient temperature, under atmospheric pressure and the beaker wasn't covered up. Thereafter, the synthesized trisodium citrate crystals was transferred in a glass container and dry in the oven for three to four hours. Noticed that before drying in the oven, it was possible to wash the previous synthesized trisodium crystals with pure-ethanol to remove water molecules and/or to refine the crystallization. Finally, white crystals of trisodium citrate salt (figure 2) was obtained and stored in a glass sealed container in a desiccator.

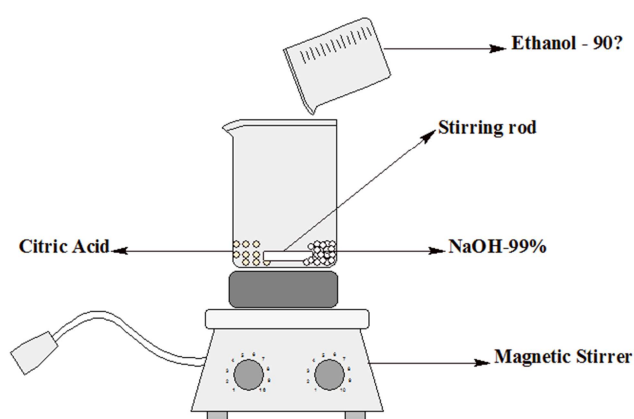


Figure 1. Synthesis of trisodium citrate.

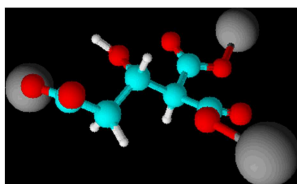


Figure 2. Trisodium citrate structure.

4. Titration Procedure of All Synthesized $C_6H_5O_7Na_3$ Crystals with Hydrochloric Acid HCl-0.1N

In continuity with what has been described previously, all synthesized trisodium citrate crystals which smelled less of alcohol-ethanol was dissolved in 35[ml] of distilled water. Then, take 2[ml] sample of this solution using a pipette-10[ml]. Dilute this sample with 15[ml] of distilled water to obtain 17[ml] of solution to be titrated. Add two or three drops of helianthine indicator solution and a stirring rod. The solution turn to orange-yellow. Put the hydrochloric acid 0.1N titration solution inside a graduated burette-50[ml] and place the previous solution to be titrated on a magnetic stirrer with moderated rotation speed. Begin to titrate with slow speed of solution titration drops and noticed that after some milliliters of hydrochloric acid 0.1N the titrated solution in the beaker-250[ml] turned to red corresponding to the equivalent point of this titration. Record the hydrochloric acid 0.1N equivalent-volume (V_e) corresponding to this equivalent-point.

Thus, the moles-quantities of Na^+ ions titrated with HCl-0.1N in the 2[ml] sample was

$$n_{Na^+}[\text{moles} - 2ml] = (0,1 \times V_e[ml] \times 10^{-3}) \quad (2)$$

The moles-quantities of Na^+ in the 35[ml] was

$$n_{Na^+}[\text{moles} - 35ml] = \frac{n_{Na^+}[\text{moles} - 2ml] \times 35}{2} \quad (3)$$

The total moles-quantities of trisodium citrate- $C_6H_5O_7Na_3$ synthesized was

$$n_{C_6H_5O_7Na_3}[\text{total moles}] = \frac{n_{Na^+}[\text{moles} - 35ml]}{3} \quad (4)$$

The molar yields of this reaction compared with the initial moles-quantities of citric acid was

$$T(\%) = \frac{n_{C_6H_5O_7Na_3}[\text{total moles}]}{n_{\text{citric acid}}[\text{total moles} - \text{Initial}]} \times 100 \quad (5)$$

5. Application of This Procedure Titration to Evaluate the Speed Constant of This $C_6H_5O_7Na_3$ Synthesis

5.1. Experimental Results and Discussions of This Trisodium Citrate- $C_6H_5O_7Na_3$ Crystals Synthesis – Conversion Evolution

Table 2. Experimental results of this trisodium citrate- $C_6H_5O_7Na_3$ crystals synthesis.

Time [s]	Conversion (%) – T	$C_6H_5O_7Na_3$ crystals color	$C_6H_5O_7Na_3$ concentrated solution [mol.l ⁻¹]	Concentrated $C_6H_5O_7Na_3$ solution refractive index
60	83.05	white	0.6914	1.3510
90	83.63			
900	89.19			
1,800	92.56			

This table 2 and figure 3 showed the citric acid conversion evolution according to the time of reaction. The synthesized trisodium citrate was white and its concentrated solution 0.6914[mol.l⁻¹] obtained by dissolving them in distilled

water had 1.3510 refractive index. Noticed that this value of refractive index was less than for the trisodium citrate di-hydrate in the bibliography (1.58) [18] certainly because of the dissolution in distilled water which decrease its

refractive index as showed in bibliography [19]. Indeed, this was taken to $1[\text{mol.l}^{-1}]$.
 refractive index increased to 1.3640 when its concentration

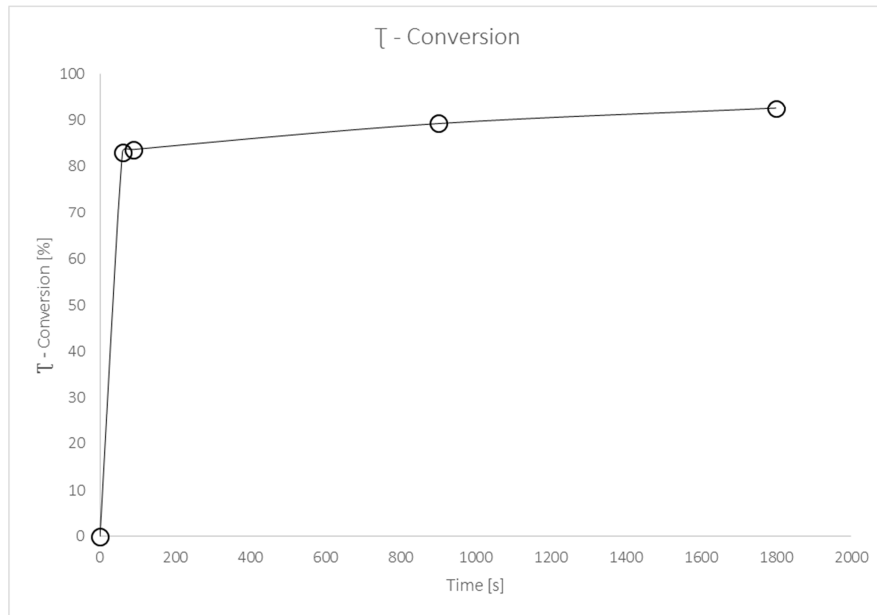
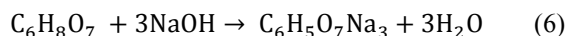


Figure 3. Conversion of citric acid molecules evolution compared with the time reaction.

The initial conversion was very higher (figure 3) and confirmed that not only this reaction is slightly endothermic [14], but also the present experimental conditions and procedure with the solvent-ethanol 90° put together were efficient to produce the trisodium citrate. Indeed, noticed firstly that the molar ratio of sodium hydroxide-NaOH divided by citric acid- $\text{C}_6\text{H}_8\text{O}_7$ was equal to 4.38 showing that the NaOH wasn't completely in excess but the initial conversion was very important as in the case of NaOH-sodium hydroxide in excess. Secondly, this higher initial conversion confirmed the role and efficacy of not only the ethanol-90° as solvent but also the efficacy of the raw materials quantities in this experimental conditions-procedure where citric acid and sodium hydroxide were initially completely soluble in the used solvent-ethanol increasing the efficacy of their collisions and also the solubility of all their remainders and eventual by-products like sodium ethoxide- $\text{C}_2\text{H}_5\text{ONa}$ soluble in the solvent ethanol.

5.2. Progress Reaction and Kinetical Studies of This Trisodium Citrate- $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ Crystals Reaction Synthesis

As previously said in §.2, the chemical equation of this trisodium citrate- $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ crystals synthesis was



Thus, speed of this reaction equals to

$$V = k \times [\text{C}_6\text{H}_8\text{O}_7]^\alpha \times [\text{NaOH}]^\beta \quad (7)$$

Yet, as discussed previously on §5.2., under this experimental conditions and according to the important initial conversion, the sodium hydroxide moles-quantities could be considered as in excess compared with citric acid

moles-quantities and assuming that the reactional volume was constant during the synthesis-reaction equal to 21[ml].

Consequently,

$$[\text{C}_6\text{H}_8\text{O}_7]^\alpha \ll [\text{NaOH}]^\beta \quad (8)$$

So, the observed speed constant was

$$V = k_{\text{NaOH}} \times [\text{C}_6\text{H}_8\text{O}_7]^\alpha \quad (9)$$

Such as

$$k_{\text{NaOH}} = k \times [\text{NaOH}]^\beta \quad (10)$$

and α the reaction order

Kinetically,

$$V = k_{\text{NaOH}} \times [\text{C}_6\text{H}_8\text{O}_7]^\alpha = + \frac{d[\text{C}_6\text{H}_5\text{O}_7\text{Na}_3]}{dt} = - \frac{d[\text{C}_6\text{H}_8\text{O}_7]}{dt} \quad (11)$$

1) Firstly, assuming that the reaction was the first order, the equation (11) became

$$V = k_{\text{NaOH}} \times [\text{C}_6\text{H}_8\text{O}_7]^1 = - \frac{d[\text{C}_6\text{H}_8\text{O}_7]}{dt} \quad (12)$$

$$\Leftrightarrow - \frac{d[\text{C}_6\text{H}_8\text{O}_7]}{[\text{C}_6\text{H}_8\text{O}_7]^1} = k_{\text{NaOH}} \times dt \quad (13)$$

Resolving this equation conducted to

$$(k_{\text{NaOH}} \times t) + K = -\text{Ln}[\text{C}_6\text{H}_8\text{O}_7] \quad (14)$$

Such as K is a constant

At initial time $t=0$, $[\text{C}_6\text{H}_8\text{O}_7] = [\text{C}_6\text{H}_8\text{O}_7]_{\text{initial}}$, so $K = -\text{Ln}([\text{C}_6\text{H}_8\text{O}_7]_{\text{initial}})$

The equation (14) became

$$(k_{\text{NaOH}} \times t) = \text{Ln}\left(\frac{[\text{C}_6\text{H}_8\text{O}_7]_{\text{initial}}}{[\text{C}_6\text{H}_8\text{O}_7]_t}\right) \quad (15)$$

In the other words, if this reaction is first order compared with citric acid- $C_6H_8O_7$, the following equation (16) must be a straight line according to time t

$$\ln\left(\frac{[C_6H_8O_7]_{initial}}{[C_6H_8O_7]_t}\right) = k_{NaOH} \times t \quad (16)$$

and its slope gave the observed speed constant k_{NaOH}

2) Secondly, assuming that the reaction was the first order, the equation (12) became

$$V = k_{NaOH} \times [C_6H_8O_7]^2 = -\frac{d[C_6H_8O_7]}{dt} \quad (17)$$

$$\leftrightarrow -\frac{d[C_6H_8O_7]}{[C_6H_8O_7]^2} = k_{NaOH} \times dt \quad (18)$$

Resolving this equation conducted to

$$(k_{NaOH} \times t) + K = \frac{1}{[C_6H_8O_7]} \quad (19)$$

Such as K is a constant

At initial time $t=0$, $[C_6H_8O_7] = [C_6H_8O_7]_{initial}$, so

$$K = \frac{1}{[C_6H_8O_7]_{initial}}$$

The equation (19), became

$$(k_{NaOH} \times t) = \left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right) \quad (20)$$

In the other words, if this reaction is second order compared with citric acid- $C_6H_8O_7$, the following equation (21) must be a straight line according to time t

$$\left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right) = (k_{NaOH} \times t) \quad (21)$$

And its slope gave the observed speed constant k_{NaOH}

The following table 3 gave the evolution of citric acid concentrations and the results of the previous equations (16) and (21) according to time.

Table 3. Experimental results of trisodium citrate- $C_6H_5O_7Na_3$ crystals reaction synthesis.

Time [s]	$[C_6H_8O_7]_{initial}$	$[C_6H_8O_7]_t$	$\ln\left(\frac{[C_6H_8O_7]_{initial}}{[C_6H_8O_7]_t}\right)$	$\left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right)$
60	0.2492	0.0422	1.7750	19.6665
90	0.2491	0.0408	1.8103	20.5164
900	0.2492	0.0269	2.2250	33.1233
1,800	0.2491	0.0186	2.5980	49.9105

Drawing the curves $\ln\left(\frac{[C_6H_8O_7]_{initial}}{[C_6H_8O_7]_t}\right)$ and $\left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right)$ evolutions (§5.1.) compared with the time reactions, the following straight line figures (Figure 4 and Figure 5) were obtained.

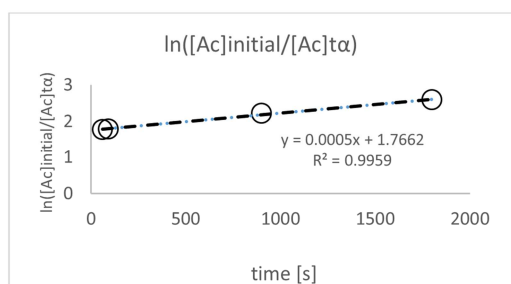


Figure 4. $\ln\left(\frac{[C_6H_8O_7]_{initial}}{[C_6H_8O_7]_t}\right)$ evolution compared with time reaction.

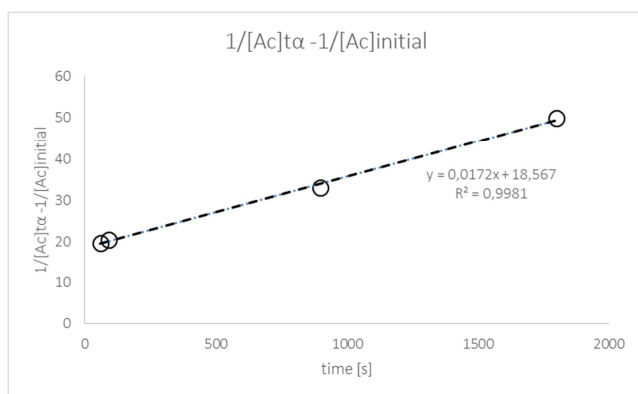


Figure 5. $\left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right)$ evolution compared with time reaction.

Seeing that the (R^2) of these figures 2 and 3 were in the vicinity of 0.997 and seeing also the conversion evolution (Figure 3) such as the moles quantities of sodium hydroxide molecules were in excess compared with citric acid molecules but noticed also that the figure 5 have an interception with the origin $O(0,0)$ higher ($R^2 = 0$) than the figure 4 ($R^2 = -12.77$); it was more adequate to affirm that according these results and the experimental conditions (Table 1), this reaction between citric acid and sodium hydroxide was second order compared with the citric acid concentration (Figure 5). This second order could explained also the role of the solvent-ethanol not only as responsible of sodium hydroxide and citric acid collisions efficacy conducting to the initial higher conversion but also its possible reactions with sodium hydroxide to give the by-products sodium ethoxide soluble in ethanol [17].

Thus, the equation (21) $\left(\frac{1}{[C_6H_8O_7]_t} - \frac{1}{[C_6H_8O_7]_{initial}}\right) = (k_{NaOH} \times t)$ was experimentally verified and the observed speed constant $k_{NaOH} = k \times [NaOH]^\beta$ (10) was equals to the slope of the figure 5 that is to say $1.72E-2 [L \times mol^{-1} \times s^{-1}]$.

Seeing that the initial sodium hydroxide concentration was $1.10 [mol.l^{-1}]$, so the speed constant was approximately $1.56E-2 [L^2 \times mol^{-2} \times s^{-1}]$.

Finally, drawing the curve showing the trisodium citrate- $C_6H_5O_7Na_3$ crystals in the reaction solution evolution with reaction time, the following figure 6 was obtained.

Noticed that, initially from zero second to 90 seconds, this concentration evolution could be equivalent to the following figure 7 from which it could be evaluated the initial concentration evolution of the synthesized trisodium citrate- $C_6H_5O_7Na_3$ crystals in the reaction solution evolution with time reaction. In the other words, the initial speed

formation of synthesized sodium ethoxide was equal to $0.0027 \text{ [mol} \times \text{l}^{-1} \times \text{s}^{-1}]$ (Figure 7).

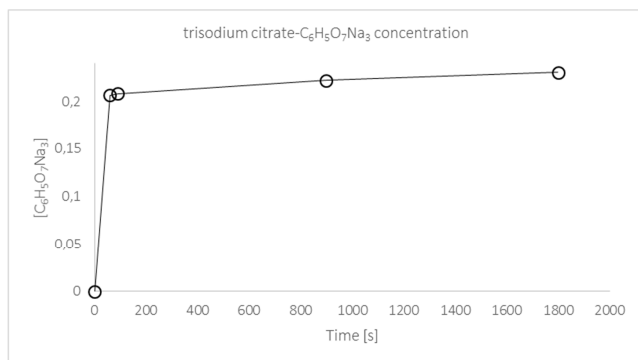


Figure 6. Concentration of trisodium citrate- $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ crystals in the reaction solution evolution compared with the time reaction.

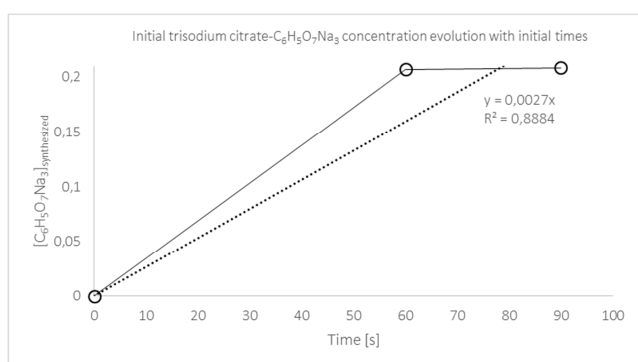


Figure 7. Initial Concentration of trisodium citrate- $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ evolution with initial time.

6. Conclusion

This synthesis of trisodium citrate crystals $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ was carried out successfully at room temperature, under normal atmospheric pressure and in the open air from sodium hydroxide-99% relatively in excess of citric acid by 4.38 twice higher and ethanol-90% permitting to synthesize excellent product like sodium hydroxide sodium in excess, thus its saving than real theoretical excess. The initial pH calculated was equal to 15.21. The refractive index of its concentrated solution with distilled water in the order of 0.6914 [mol/L] was 1.3510 slightly less than for the trisodium citrate di-hydrate in the bibliography (1.58) [18] certainly because of the dissolution in distilled water which decrease its refractive index as showed in bibliography [19]. The established procedure for titrating trisodium citrate with 0.1N hydrochloric acid was successful and allowed to plot the curve of the conversion as a function of time and also to establish kinetic parameters as a function of these experimental conditions such that the reaction between citric acid and sodium hydroxide is citric acid second order with respect to the concentration of citric acid. The speed constant of this reaction synthesis of trisodium citrate- $\text{C}_6\text{H}_5\text{O}_7\text{Na}_3$ crystals under these previous experimental conditions was equal to $1.56\text{E-}2 \text{ [L}^2 \times \text{mol}^{-2} \times \text{s}^{-1}]$. Also, the initial speed

formation of synthesized sodium ethoxide was equal to $0.0027 \text{ [mol} \times \text{l}^{-1} \times \text{s}^{-1}]$ and the citric acid conversion only after 30 minutes of reaction was 92.56%. To estimate the porosity of these trisodium citrate crystals, 0.0174 [g] was immersed in 15 [ml] of pure isopropyl alcohol using a crystallizer covered a bell jar and a beaker-250[ml] to avoid humid air condensation for 150 [min] [17]. Thus, its porosity value was approximately 48.67% with density equal to 1.79 [g/ml] . Noticed that during this porosity estimation using pure isopropyl alcohol, pay attention to the final weight of filled-sample which corresponded to the status such as the crystallizer was dry except just underneath every trisodium citrate crystals bonded against the crystallizer. As said previously and according to many literatures [20-22], this synthesis was used in various applications areas including synthesis in laboratory of citric acid metal-alkoxide [20-22].

Acknowledgements

Sincere thanks to the E. S. P. A Polytechnics. And, sincere respect to Chemical Process Engineering Department (E. S. P. A) as well as Chemical Engineering Laboratory staff.

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