

Research/Technical Note

Sodium Ethoxide Concentrated Solution Synthesis at Ambient Temperature Using Sodium Hydroxide and Ethanol-90 in Excess

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Abstract: Sodium ethoxide is a specific chemical product of synthesis used in laboratory, thus its presence at the suppliers stays very periodic and on order. It was in that sense that this project to synthesize a concentrated solution of sodium ethoxide got off at ambient temperature, under normal atmospheric pressure in an uncovered beaker using ethanol 90% of purity and sodium hydroxide 99% of purity seeing that the ethanol molecules were in excess in comparison with sodium hydroxide molecules which quantities were all soluble in ethanol. The initial calculated pH was equal to 15.23. In addition, not only an extraction procedure of the concentrated sodium ethoxide was done but also a titration procedure of the concentrated sodium ethoxide solution by hydrochloric acid HCl-0.1N was established and permitted to evaluate kinetic parameters of this synthesis reaction on these experimental conditions. The evaluated molar conversion from the initial quantities of sodium hydroxide was 88.49% after 3h25mn of reaction. It was noticed that the formation of sodium ethoxide was initially very important and very speed confirming the negativity of its standard molar enthalpies. Thus, the initial speed formation of synthesized sodium ethoxide was approximately equal to $0.013 \text{ [mol} \times \text{l}^{-1} \times \text{s}^{-1}]$. Once synthesized, the total crystallization of the concentrated sodium ethoxide was tried with an evaluation of its porosity by immersion in dichloromethane giving a value in the order of 54.60%.

Keywords: Sodium Ethoxide, Ethanol, Sodium Hydroxide, Hydrochloric Acid

1. Introduction

In the first time, laboratory synthesis of concentrated solution of sodium ethoxide was done using an excess of ethanol (90% of purity) in comparison with sodium hydroxide which were completely soluble in the used ethanol volume. Laboratory materials and chemicals used during this synthesis were beaker-250ml, water bath Büchi B-480, rotavapor Büchi, magnetic stirrer-Fischer scientific, proofer, crystallizer, syringe, test tube-100ml, test tube-50ml, burette-50ml, thymol

blue indicator dye, sodium hydroxide 99%, ethanol 90%. Then, the amount of sodium hydroxide molecules of the concentrated solution was determined by titration with hydrochloric acid HCl-0.1N using a burette-50ml and indicator dye thymol blue according to a procedure described afterwards. Finally, a total crystallization of the synthesized concentrated solution of sodium ethoxide was tried and the crystallized sodium ethoxide porosity was evaluated by submerging 0.0174 [g] in 15 [ml] of dichloromethane during 150 minutes.

2. Experimental Conditions of Sodium Ethoxide- C_2H_5ONa Synthesis

The raw materials used to synthesize the sodium ethoxide (C_2H_5ONa) were ethanol 90° and sodium hydroxide 99% (NaOH) such as the ethanol was in excess compared to sodium ethoxide. Indeed, as shown in the following table 1, the molar ratio of ethanol divided by sodium hydroxide-NaOH was equal to 5.18. On this experimental conditions, all used NaOH-raw material were soluble in C_2H_5OH -90°-raw

material during synthesis seeing that the sodium hydroxide solubility in ethanol was 139 [g/l] [1-5], so the raw materials collisions certainly increased efficiently. In addition, the calculated pH of the solution were 15.23 and the recorded pH of the solution after one minute of mixing were 11.48 seeing that it was composed with two strong bases such as the ethanol was in excess compared to sodium ethoxide [6-7]. Noticed that this pH=11.48 of the solution only after one minute of reaction (Table 1) was already in the vicinity of a sodium ethoxide-20% solution in ethanol pH for synthesis [8].

Table 1. Experimental conditions of sodium ethoxide synthesis using ethanol-90° and sodium hydroxide.

Raw materials	Ethanol- C_2H_5OH -90°	sodium hydroxide-NaOH-99%
Initial volume [ml]	93.33	-
Weight [g]	-	11.22
Raw materials quantities [moles]	1.4394	0.2778
Initial raw materials concentrations [mol.l ⁻¹]	15.4227	2.9765
Raw materials molar ratio of ethanol divided by sodium hydroxide	5.18	1.00
Reaction temperature	Ambient temperature	
Reaction durations [s]	90 – 2,400 – 11,700	
Sodium hydroxide-NaOH soluble in ethanol [g]	11.676	
Calculated - pH	15.23	
Recorded pH after one minute of reaction	11.48	

3. Experimental Procedure of Sodium Ethoxide- C_2H_5ONa Synthesis

First of all, weight the used sodium hydroxide-11.22 [g] with a precision balance, put it into a beaker-250 [ml] in which a stirring rod was placed. Then, measure the volume-93.33 [ml] of ethanol used with 500 [ml] and 50 [ml] test tubes. Put the beaker-250 [ml] on a magnetic stirrer, begin to stir slowly the sodium hydroxide 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 was dissolved after roughly thirty seconds. After approximately one minute, the solution temperature increased suddenly and the volume decreased in the vicinity of 92 [ml]. These results confirmed the negativity of its standard molar enthalpies equals to -413.39 ± 1.45 [Kj.mol⁻¹] [9-10] seeing that the synthesis of sodium ethoxide using NaOH and alcohol-ROH ($R = -C_3H_7$, $-C_4H_9$, $-C_5H_{11}$) was done by Vacek et al. at 1984 [11]. 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 homogeneous oily solution like a detergent was obtained above with a constant refractive index 1.3765 superior than the refractive index at 20°C of the ethanol solution (100% concentration – w/w(%)) equals to 1.3614 at 100- w/w(%), 1.3425 at 14- w/w(%) and 1.3652 at 70-w/w(%) [12]. This oily solution slightly yellow in contact with a paper was a concentrated solution of sodium ethoxide- C_2H_5ONa synthesized which density was 0.88 [g.cm⁻³] [8]. Once synthesized, recorded the synthesized volume ($V_{C_2H_5ONa}$) which amount of sodium ethoxide was defined by titration with hydrochloric acid 0.1N inspired by bibliography [13]. Noticed that all the synthesis was at

ambient temperature, under atmospheric pressure and the beaker wasn't covered up. After titration, the synthesized solution of sodium ethoxide- C_2H_5ONa was transferred in a glass sealed container, then maintained, decanted and crystallized in a freezer to increase the separation between water/non-transformed NaOH (at the bottom) and sodium ethoxide- C_2H_5ONa synthesized (upstairs) during approximately 24 hours. It was noticed that the solution became more slimy with formation of little sodium ethoxide- C_2H_5ONa crystals and may be eventually little unreacted NaOH crystals at the bottom. Finally, the slimy concentrated sodium ethoxide- C_2H_5ONa solution was transferred and separated from the bottom solution with the rest of the NaOH. The synthesized white slimy concentrated sodium ethoxide- C_2H_5ONa solution was stored in a freezer.

4. Titration Procedure of Sodium Ethoxide- C_2H_5ONa Solution with Hydrochloric Acid 0.1N

As said previously, the synthesized sodium ethoxide solution was decanted over a water bath less than 288.15°C to freeze and to stop the reaction during 15 minutes. Then, take 2 [ml] sample of synthesized sodium ethoxide solution using a pipette-10 [ml] and paying attention to extract this sample in the middle of the solution synthesized in the beaker-250 [ml]. Dilute this sample with 30 [ml] of distilled water to obtain 32 [ml] of solution to be titrated. Take and put 10 [ml] of this diluted sample in a beaker-250 [ml] and add two or three drops of thymol blue indicator solution and a stirring rod. The solution turn to deep blue. 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 purplish-blue, then sparkling blue and finally suddenly yellow when the pH of the titrated solution was in the vicinity of 7 corresponding to the equivalent point of this titration. Record the hydrofluoric acid 0.1N equivalent-volume (V_e) corresponding to this equivalent-point.

Thus, the moles-quantities of sodium ethoxide- C_2H_5ONa in the 10 [ml] sample was

$$n_{C_2H_5ONa}[moles - 10ml] = (0,1 \times V_e[ml] \times 10^{-3}) \quad (1)$$

The moles-quantities of sodium ethoxide- C_2H_5ONa in the 32 [ml] was

$$n_{C_2H_5ONa}[moles - 32ml] = \frac{n_{C_2H_5ONa}[moles-10ml] \times 32}{10} \quad (2)$$

The concentration of sodium ethoxide- C_2H_5ONa in the synthesized solution was

$$[C_2H_5ONa] = \frac{n_{C_2H_5ONa}[moles-32ml]}{(2 \times 10^{-3})} \quad (3)$$

The total moles-quantities in the synthesized volume $V_{C_2H_5ONa}$ was

$$n_{C_2H_5ONa}[total\ moles - V_{C_2H_5ONa}] = [C_2H_5ONa] \times V_{C_2H_5ONa} \quad (4)$$

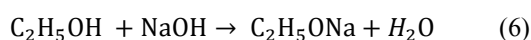
The molar yields of this reaction compared with the initial moles-quantities of NaOH was

$$T(\%) = \frac{n_{C_2H_5ONa}[total\ moles - V_{C_2H_5ONa}]}{n_{NaOH}[total\ moles - Initial]} \times 100 \quad (5)$$

5. Application of Previous Titration to Evaluate the Speed Constant of This Sodium Ethoxide- C_2H_5ONa Synthesis

5.1. Progress Reaction and Kinetical Studies of This Sodium Ethoxide- C_2H_5ONa Solution Reaction Synthesis

As previously said in the introduction, the chemical equation of this sodium ethoxide- C_2H_5ONa solution synthesis was



Thus, speed of this reaction equals to

$$V = k \times [C_2H_5OH] \times [NaOH] \quad (7)$$

Yet, under this experimental condition, the ethanol moles-quantities was in excess compared with sodium hydroxide moles-quantities and assuming that the reactional volume was constant during the synthesis-reaction. Indeed, after 90 [s] of reactions the volume stayed at 92 [ml] and after 11,700 [s] of reactions stirred without lid still stayed at 87.5 [ml] confirming the formation of a concentrated sodium

ethoxide- C_2H_5ONa solution above. Therefore, during this speed constant evaluation, it was assumed that the reactional volume was constant and equals to 92 [ml] assuming that initially 1.3 [ml] of alcohol was evaporated.

Consequently,

$$[NaOH] \ll [C_2H_5OH] \quad (8)$$

So, the observed speed constant was

$$V = k_{C_2H_5OH} \times [NaOH]^a \quad (9)$$

Such as

$$k_{C_2H_5OH} = k \times [C_2H_5OH] \quad (10)$$

and α the reaction order

Kinetically,

$$V = k_{C_2H_5OH} \times [NaOH]^a = + \frac{d[C_2H_5OH]}{dt} = - \frac{d[NaOH]}{dt} \quad (11)$$

As previously mentioned, it was assumed that this reaction synthesis occurred at constant reactional volume to facilitate its approximate speed constant calculations. Thus,

$$V = k_{C_2H_5OH} \times \frac{n_{NaOH}^a}{V^a} = + \frac{dn_{C_2H_5OH}}{Vdt} = - \frac{dn_{NaOH}}{Vdt} \quad (12)$$

The following table 2 showed the progress of this reaction,

Table 2. Reaction progress of sodium ethoxide synthesis.

Time - t	C_2H_5OH	NaOH	C_2H_5ONa
t = 0 (initial)	n_{Ci}	n_{Ni}	0
t = t_α	$n_{Ci} - \alpha$	$n_{Ni} - \alpha$	α

Such as α was the moles quantities of sodium ethoxide formed at time t_α

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

$$V = k_{C_2H_5OH} \times \frac{n_{NaOH}^1}{V^1} = + \frac{dn_{C_2H_5OH}}{Vdt} = - \frac{dn_{NaOH}}{Vdt} \quad (13)$$

Compared with NaOH (13) became

$$V = k_{C_2H_5OH} \times n_{NaOH} = - \frac{dn_{NaOH}}{dt} \quad (14)$$

and at the time t_α

$$V = k_{C_2H_5OH} \times (n_{Ni} - \alpha) = - \frac{d(n_{Ni} - \alpha)}{dt} \quad (15)$$

Incorporate $X = (n_{Ni} - \alpha)$ so

$$k_{C_2H_5OH} \times dt = - \frac{dX}{X} \quad (16)$$

Resolving this equation conducted to

$$(k_{C_2H_5OH} \times t) + K = -\ln X \quad (17)$$

Such as K is a constant

At initial time $t=0$, $\alpha=0$. So, $X=n_{Ni}$

and $K = -\ln(n_{Ni})$

The equation (17) became

$$(k_{C_2H_5OH} \times t) = -\ln(n_{Ni}-\alpha) + \ln(n_{Ni}) \quad (18)$$

In the other words, if this reaction is first order compared with sodium hydroxide-NaOH, the following equation (19) must be a straight line according to time t

$$\ln\left(\frac{n_{Ni}}{n_{Ni}-\alpha}\right) = k_{C_2H_5OH} \times t \quad (19).$$

Seeing that α which was the moles quantities of sodium ethoxide formed at time t_α equals to

$$\alpha = [T(\text{conversion}\%) \times n_{Ni}].$$

Thus, the equation (19) became

$$\ln\left(\frac{1}{1-T}\right) = k_{C_2H_5OH} \times t \quad (20)$$

and its slope gave the observed speed constant $k_{C_2H_5OH}$

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

$$V = k_{C_2H_5OH} \times \frac{n_{NaOH}^2}{V^2} = + \frac{dn_{C_2H_5OH}}{Vdt} = - \frac{dn_{NaOH}}{Vdt} \quad (21)$$

Compared with NaOH (20) became

$$V = k_{C_2H_5OH} \times \frac{n_{NaOH}^2}{V^2} = - \frac{dn_{NaOH}}{Vdt} \quad (22)$$

and at the time t_α

$$V = k_{C_2H_5OH} \times \frac{(n_{Ni}-\alpha)^2}{V^2} = - \frac{d(n_{Ni}-\alpha)}{Vdt} \leftrightarrow \quad (23)$$

$$V = k_{C_2H_5OH} \times (n_{Ni}-\alpha)^2 = -V \times \frac{d(n_{Ni}-\alpha)}{dt} \quad (24)$$

Incorporate $X=(n_{Ni}-\alpha)$ so

$$k_{C_2H_5OH} \times dt = -V \times \frac{dX}{X^2} \quad (25)$$

Resolving this equation conducted to

$$(k_{C_2H_5OH} \times t) + K = + \frac{V}{X} \quad (26)$$

Such as K is a constant

At initial time $t=0$, $\alpha=0$. So, $X=n_{Ni}$ and $K = + \frac{V}{n_{Ni}}$

The equation (26) became

$$(k_{C_2H_5OH} \times t) + \frac{V}{n_{Ni}} = + \frac{V}{(n_{Ni}-\alpha)} \quad (27)$$

In the other words, if this reaction is second order compared with sodium hydroxide-NaOH, the following equation (28) must be a straight line according to time t

$$(k_{C_2H_5OH} \times t) = + \frac{(\alpha \times V)}{(n_{Ni}-\alpha) \times n_{Ni}} \quad (28)$$

And its slope gave the observed speed constant $k_{C_2H_5OH}$

Table 3. Experimental results of this sodium ethoxide- C_2H_5ONa solution reaction synthesis.

Time [s]	Conversion (%) – T At volume constant (§5.1.)	Concentrated C_2H_5ONa solution refractive index	C_2H_5ONa solution concentration [mol.l ⁻¹]	C_2H_5ONa solution color	n_{Ni} - NaOH [moles]	$\ln\left(\frac{1}{1-T}\right)$	$\frac{(T \times V)}{(n_{Ni} - T) \times n_{Ni}}$
90	38.69	1.3765	1.17	white	0.2778	0.4891	0.2090
2400	85.88	1.3745	2.62	white	0.2806	1.9573	1.9934
11700	88.49	1.3765	2.67	white	0.2778	2.1619	2.5460

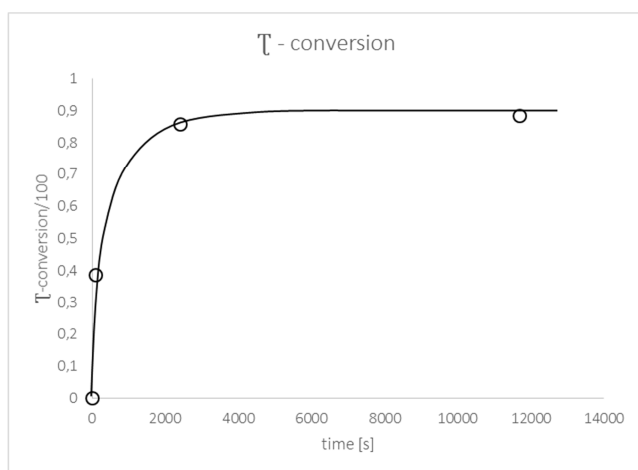


Figure 1. Conversion of sodium hydroxide-NaOH evolution compared with the time reaction.

5.2. Experimental Results and Discussions of This Sodium Ethoxide- C_2H_5ONa Solution Reaction Synthesis – Speed Constant Determination

The initial conversion was higher (Figure 1) and confirmed

not only the negative value of sodium alkoxides formation standard molar enthalpies [9-10] and consequently the observed exothermic and instantaneous of reaction between the raw materials NaOH and alcohol-ethanol in excess but also the efficacy of the experimental pH which was calculated in the vicinity of 15.23 and observed at 11.48 after one minute of reaction. Drawing the curves of $\ln\left(\frac{1}{1-T}\right)$ and $\frac{(\alpha \times V)}{(n_{Ni}-\alpha) \times n_{Ni}}$ evolutions (§5.1.) compared with the time reactions, the following figures (Figure 2 and Figure 3) were obtained.

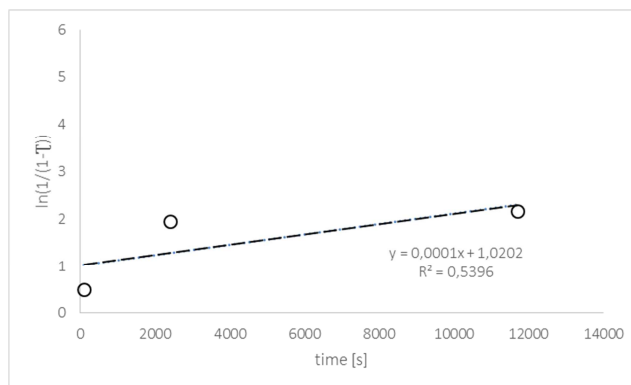


Figure 2. $\ln\left(\frac{1}{1-T}\right)$ evolution compared with time reaction.

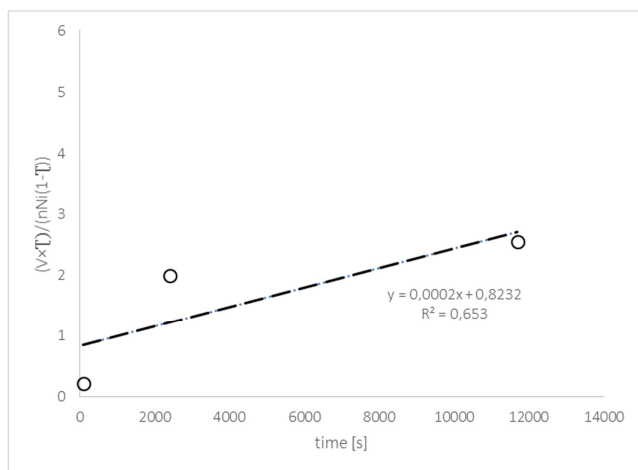


Figure 3. $\frac{(\alpha \times V)}{(n_{Ni} - \alpha) \times n_{Ni}}$ evolution compared with time reaction.

Seeing that the (R^2) of these figures 2 and 3 were in the vicinity of 0.60 and seeing also the conversion evolution (Figure 1) such as the moles quantities of solvent-reagent ethanol molecules were in excess compared with sodium hydroxide molecules but noticed that the figure 3 have an interception with the origin $O(0,0)$ higher ($R^2 = 0.2922$) than the figure 2 ($R^2 = -0.453$); it was more adequate to affirm that according these results and the experimental conditions (Table 1), this reaction between ethanol and sodium hydroxide was second order compared with the sodium hydroxide concentration (Figure 2). Thus, the equation (28) $(k_{C_2H_5OH} \times t) = + \frac{(\alpha \times V)}{(n_{Ni} - \alpha) \times n_{Ni}}$ was verified and the observed speed constant $k_{C_2H_5OH} = k \times [C_2H_5OH]$ (10) was equals to the slope of the figure 3 that is to say $2E-4 \text{ [mol} \times \text{L}^{-1} \times \text{s}^{-1}]$. Seeing that the initial ethanol concentration was $15.67 \text{ [mol.l}^{-1}]$, so the speed constant was approximately $1.28E-5 \text{ [s}^{-1}]$.

Finally, drawing the curve showing the sodium ethoxide solution concentration evolution with reaction time, the following figure 4 was obtained.

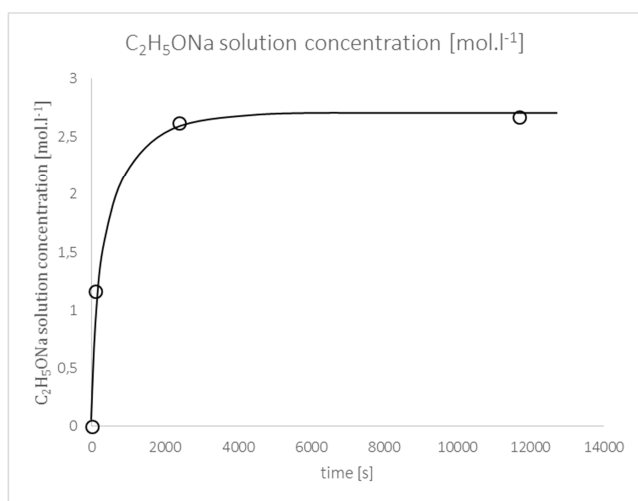


Figure 4. Concentration of sodium ethoxide solution evolution compared with the time reaction.

Noticed that, initially from zero second to 90 seconds, this concentration evolution could be equivalent to the following figure 5 from which it could be evaluated the initial concentration evolution of the synthesized sodium ethoxide in the solution with time reaction. In the other words, the initial speed formation of synthesized sodium ethoxide was equal to $0.013 \text{ [(mol} \times \text{l}^{-1} \times \text{s}^{-1}]$.

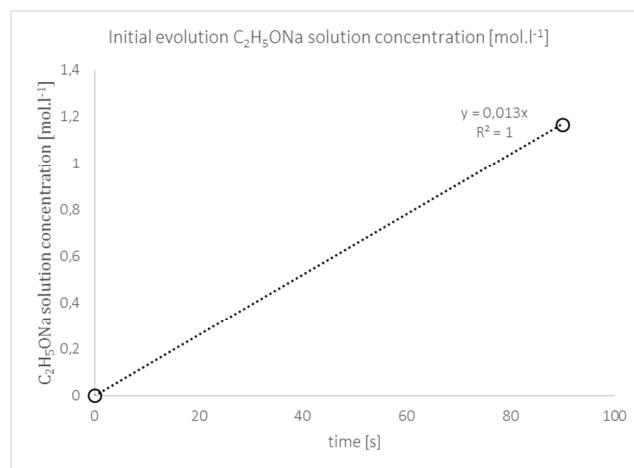


Figure 5. Initial evolution of sodium ethoxide concentration solution compared with the time reaction.

6. Conclusion

The synthesis of the concentrated solution of sodium ethoxide in the order of $2.6 \text{ [mol.l}^{-1}]$ with 1.3765 refractive index was performed at ambient temperature, under normal atmospheric pressure and in open air from sodium hydroxide 99% and ethanol 90% in excess such as the molar ratio between these two raw materials were 5.18 and the initial calculated pH was equal to 15.23. The titration procedure of the sodium ethoxide using hydrochloric acid 0.1N was successful and permitted not only to draw the conversion evolution curve in accordance with time and but also to establish this synthesis kinetic parameters according to these experimental conditions such as the sodium hydroxide partial order was equals to one, the speed constant was equal to $1.28 \times 10^{-5} \text{ [s}^{-1}]$. Finally, the total crystallization of the concentrated sodium ethoxide solution was tried in which instead of directly put under a rotavapor to remove ethanol, this solution was heated and agitated in a water bath at $323.15 \text{ [}^\circ\text{K}]$ during 3h30minutes in open air; it turned to yellow then brown with just maximally 2 [ml] volume diminution and its refractive index stayed 1.3760. Thereafter, it was stored with a hermetic box in a freezer for 16 hours and only now it was evaporated under pressure at $313.15 \text{ [}^\circ\text{K}]$ using a rotavapor to remove ethanol. White and brown sodium ethoxide crystals were obtained and then dehydrated in a proofer during at least 15 hours. In addition, the sodium ethoxide crystals were stored in the proofer at $333.15 \text{ [}^\circ\text{K}]$ during 14 days and their color became increasingly white. To estimate the porosity of this very hygroscopic sodium ethoxide crystals increasingly devoid of ethanol [14], 0.0174 [g] was submerged in 15 [ml]

of dichloromethane using a crystallizer covered with a beaker-250 [ml] to avoid humid air condensation during 150 [mn]. Thus, its porosity value was 54.60% with very low density and apparent density respectively in the order of 0.25 [g/ml] and 0.4885 [g/ml] [14-17].

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