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Preparation of sodium-meta borate with series reactor and laboratory conditions

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Abstract
Sodium per-borate is obtained from the reaction between sodium-meta borate and hydrogen peroxide in the presence of magnesium sulfate or magnesium chloride as a stabilizer. Sodium-meta borate is obtained from reaction between sodium hydroxide and borax in the presence of sodium silicate. Typically this reaction is performed in a batch reactor. In this study, first, the reaction rate constant between sodium hydroxide and borax is obtained 4.94*10 $(dm^3/mole)^{2.12}(sec)^{-1}$, the reaction degree for sodium hydroxide was 2.47 and the reaction degree for borax was 0.65. Then continue using the obtained data in three series reactors with a volume of 1000ml for each operating conditions, respectively conversion percentage were in first reactor 41.6, second reactor 48.78 and the third reactor 50.96 while the percentage of computational conversion was respectively 84.79, 88.34 and 94.08. For obtaining the percentage of conversion in larger reactors respectively 1000, 1042 and 1110.

Keywords: sodium per-borate, sodium-meta borate, borax, sodium hydroxide, series reactors

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INTRODUCTION

Bleaching agents in detergent are divided in two categories:

1) Chlorine compounds
2) Peroxide bleaching agents (peroxides)

Peroxide compounds are included hydrogen peroxide, sodium per-carbonate and sodium per- borate. With the presence of these compounds in detergents they can provide bleaching and spotlighting (Rahbar 2006). They will be easier in storage and using for solid state of sodium per-carbonate and sodium per-borate (Rahbar 2005).

Sodium per-borate obtained from the reaction between sodium-meta borate and hydrogen peroxide in the presence magnesium sulfate or magnesium chloride. The reaction temperature is about 10°C and also sodium-meta borate prepared from sodium hydroxide and borax in about 50°C and sodium silicate used as a stabilizer (Mandar and Pangarkor 2003). These reactions are carried out in the batch reactor (Mandar and Pangarkor 2003). The conditions for the preparation of sodium-meta borate are such that the reaction temperature is 50°C and in the batch reactor and soluble state. The reaction between sodium hydroxide and borax carried out in a dry condition, solid state and high temperature also be performed (Piskin et al. 2012). Major work done in the preparation of sodium per-borate to improve the condition nucleation and core growth which is the reaction sodium-meta borate and hydrogen peroxide (Vrhunes 1999).

In this study focus is on the preparation sodium-meta borate in solution conditions. Sodium-meta borate prepared in batch reactor and in this study tried to get this compound in continuous conditions and high efficiency.

First the equation of reaction rate and related parameters in the batch reactor were determined by PH metric method. Then according to these data, the conditions of sodium-meta borate production in series and continuous reactors were investigated and compared from the empirical data results. In this conditions it can be estimated that sodium-meta borate is obtained with continuous conditions and improved that. To obtain equation of reaction rate a reaction in homogenous system it use a batch reactor. It obtained changes concentration-time and it can obtained with this conditions degree reaction for each component and reaction rate constant. To achieve this target it can use methods such as differential, integral, initial speed, half-life, liner least squares analysis, non-liner least squares analysis. Differential method will be for fixed volume system and changes concentration-time (Fogler 2002):

$$\frac{-dC_A}{dt} = kC_A^{\alpha}$$

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It will be with logarithm of this equation:

$$\ln\left(\frac{-dC_A}{dt}\right) = \ln k + \alpha \ln C_A$$

By drawing $\ln\left(\frac{-dC_A}{dt}\right)$ in terms of $\ln C_A$ on paper ln-ln line slope was for degree reaction and it obtained reaction rate constant from to use formula:

$$k = \frac{-dC_A/dt}{C_A^\alpha}$$

The numerical differential method is used to determine $\frac{-dC_A}{dt}$ values when the concentration is obtained in the same time interval.

$$t_1 - t_0 = t_2 - t_1 = \Delta t$$

The differential values $\frac{dC_A}{dt}$ is obtained from three-point differential equation. These equations are shown below:

The initial point (Fogler 2002):

$$\left[\frac{dC_A}{dt}\right]_{t_0} = \frac{1}{2\Delta t}\left[-3C_{A_0} + 4C_{A,i} - C_{A,n}\right]$$

The middle point:

$$\left[\frac{dC_A}{dt}\right]_{t,i} = \frac{1}{2\Delta t}\left[C_{A(i+1)} - C_{A(i-1)}\right]$$

The final point:

$$\left[\frac{dC_A}{dt}\right]_{t,n} = \frac{1}{2\Delta t}\left[C_{A(n-2)} - 4C_{A(n-1)} + 3C_{A,n}\right]$$

The desired reaction is in this study:

$$Na_2B_4O_7 + 2NaOH \rightarrow 4NaBO_2 + H_2O$$

The equation of reaction rate is shown follow (Fogler 2002):

$$-r_A = kC_A^\alpha . C_B^\beta$$

A for $Na_2B_4O_7$ and B for $NaOH$.

NaOH is limiting reactant in this reaction and it is considered different concentration from that and with used PH meter method, it is recorded pH and the results are as follows. The initial concentration of sodium hydroxide choose at the lowest level and concentration of borax was considered higher than it until it assume fixed concentration of borax. In this way, the equation of reaction rate is shown following:

$$-r_A = k'C_B^\beta$$
$$k' = kC_A^\alpha$$

k', β is obtained in the first stage then the sodium hydroxide concentration is increased, the borax concentration stays constant and k,α is obtained, relation of speed is obtained with this conditions. By setting the values in the relationship of calculated the reactor volume, any change in reactor conditions can be made (Fogler 2002):

$$V = F_{A_0}\left(\frac{1}{-r_A}\right)x$$

Experimental results were compared with calculated values and using that conversion percentage can go up. This work can be done with two way:

- First, several continuous reactor were used
- Second, one big reactor were used

Table 1. The results pH for determine β

time	pH	Concentration of OH^-
0	9.76	$5.754 * 10^{-5}$
1	9.71	$5.129 * 10^{-5}$
2	9.68	$4.786 * 10^{-5}$
3	9.66	$4.571 * 10^{-5}$
4	9.65	$4.467 * 10^{-5}$
5	9.64	$4.4266 * 10^{-5}$
6	9.63	$4.169 * 10^{-5}$
7	9.62	$4.074 * 10^{-5}$
8	9.62	$4.074 * 10^{-5}$
9	9.62	$4.074 * 10^{-5}$
10	9.61	$3.961 * 10^{-5}$
11	9.61	$3.961 * 10^{-5}$
12	9.61	$3.961 * 10^{-5}$

For calculating conversion value in the next reactor it can used from relationship follows (Fogler 2002):

$$V = F_{A_0}\left(\frac{1}{-r_A}\right)(x_n - x_{n-1})$$

The results are presented in conclusion.

EXPERIMENTAL

1) calculate α, β, k in relationship speed between sodium hydroxide and borax
2) calculate conversion percentage in continuous reactor and testing in continuous conditions

Experiment Determination of α, β, k

Materials and instrument for testing

Disodium tetra borate (borax) (purity 98%) with merck code 1.06306.1000, sodium hydroxide (purity 99-100) with merck code 1.06469.1000, sodium silicate (purity 40%) with merck code 1.05621.2500, Metrohm - 744 PH Meter.

Experiments for determination of α, β, k

This stage is included of:

At first, they prepared two solution from sodium hydroxide with concentration 0.04 mol/lit and borax with concentration 0.2 mol/lit, then they entered to a batch reactor by employing pH meter method and while mixing the solution in the present sodium silicate with the amount 0.1gr for stabilizer, it record changes PH, therefor concentration of OH^- will be determine. It will be obtained by used numerical differential method $\frac{-dC_B}{dt}$ and they get logarithm from $\frac{-dC_B}{dt}$, C_B and to draw the curve on the logarithmic paper it can obtain line slope, that is the same as NaOH degree reaction, also it use from equation follows the results in this stage are obtained to **Table 1** and **2** and by determination of logarithm of plot (ln $-dC_B/dt$ on y-axis and lnC_B on x-axis) was constracted:

$$-dC_B/dt = k'C_B^\beta$$
$$\ln -dC_B/dt = \ln k' + \beta \ln C_B$$

Table 2. The results for calculate logarithm C_{B_0} and $(-dC_B/dt)_0$

number	$\ln C_{B_0}$	$\ln(-dC_B/dt)_0$
1	-9.263	-16.567
2	-9.878	-17.026
3	-9.947	-17.966
4	-9.993	-18.136
5	-10.016	-18.181
6	-10.062	-18.204
7	-10.085	-18.644
8	-10.108	-18.665

Table 3. The results pH for determine α and k

time	pH	Concentration of OH^-	x	Concentration of $Na_2B_4O_7$ $N_A = N_{A_0} - 1/2\, N_{B_0} x$
0	10.96	$9.12 * 10^{-4}$	0	0.1
1	10.77	$5.888 * 10^{-4}$	0.3544	0.099838
2	10.75	$5.623 * 10^{-4}$	0.3834	0.099825
3	10.72	$5.248 * 10^{-4}$	0.4246	0.099806
4	10.65	$4.467 * 10^{-4}$	0.5102	0.099760
5	10.58	$3.802 * 10^{-4}$	0.5831	0.099730
6	10.53	$3.388 * 10^{-4}$	0.6285	0.099713
7	10.50	$3.162 * 10^{-4}$	0.6533	0.099702
8	10.49	$3.09 * 10^{-4}$	0.6612	0.099698

Table 4. The results for calculate logarithm C_{A_0} and $(-dC_A/dt)_0$

number	$\ln C_{A_0}$	$\ln(-dC_A/dt)_0$
1	-2.302	-13.14
2	-2.304	-14.13
3	-2.304	-15.894
4	-2.3045	-15.12
5	-2.3049	-14.964
6	-2.3053	-15.446
7	-2.3054	-15.964
8	-2.3055	-15.93

In order to calculate, k in another reaction, the amount of sodium hydroxide was increased to 0.4mol/lit and borax with concentration 0.2mol/lit. Conditions of reaction in the batch reactor was provided like previous reaction. pH values were measured in different times and OH^- concentration was calculated, the results in this stage are obtained to **Table 3** and **4**. Then equation below:

$$\ln -dC_A/dt/C_B^{\beta} = \ln k + \alpha \ln C_B$$

Is used to draw a curve and by determining of logarithm of plot (ln $-dC_A/dt/C_B^{\beta}$ on y-axis and lnC_A on x-axis) was constructed. By calculating the line slope, α was determined. Then reaction rate constant was obtained from the $k = -dC_A/dt/{C_A}^{\alpha}$ equation. The results brought at section results (**Table 3** and **Table 4**).

Experiments for reaction in series reactors conditions and continuous conditions

With in hand valid values for reaction speed to produce sodium-meta borate and use of a jacket glass reactor available in water 53-54°C and input flow for sodium hydroxide solution12gr/lit and borax solution15.39gr/lit for each one 0.56 ml/sec overall 1.12ml/sec and usage propeller impeller three blade, solution temperature 50°C, output flow 1.12 ml/sec (Shafiei et al. 2018). Output of the first reactor entered into the second reactor. Then the second reactor output entered the third reactor which input flow and out flow were 1.12ml/lit the results are shown in **Tables 5** and **6**.

Table 5. The results for compare between Calculate conversion percentage and Obtained

Test stage	Input flow	Reactor volume	Calculate conversion percentage	Obtained conversion percentage in test conditions	The volume need for empirical conversion percentage
1	1.12ml/sec	1000ml	84.79	41.6	542ml
2	1.12ml/sec	1000ml	88.34	48.78	610ml
3	1.12ml/sec	1000ml	94.08	50.96	710ml

Table 6. The results for compare between Reactor volume and the volume need for calculate conversion percentage

Test stage	Input flow	Reactor volume	Calculate conversion percentage	Obtained conversion percentage in test conditions	The volume need for calculate conversion percentage
1	1.12ml/sec	1000ml	84.79	41.6	1000ml
2	1.12ml/sec	1000ml	88.34	48.78	1042ml
3	1.12ml/sec	1000ml	94.08	50.96	1110ml

RESULTS AND DISCUSSION

According to the results in empirical conditions and continuous conditions values that are shown in **Table 1** and reaction rate constant value compatibility 4.94*10 $(dm^3/mole)^{2.12}(sec)^{-1}$ and reaction degree for sodium hydroxide is 2.47 and reaction degree for borax is 0.65. In order to increase the percentage of conversion in continuous production conditions, it can be done in two ways:

1) production within serial reactors
2) Increasing the reactor volume to produce with the desired conversion percentage.

By using the information on the velocity relation in the batch reactor is obtained and use them in calculating the volume for a continuous reactor with 1000ml volume, the amount of conversion percentage was 84.79. If the reactor is considered the same as the volume previous in a row, the output of the first reactor is the second input of the reactor. The conversion percentage calculating was 88.34 and for the third reactor under the same conditions was 94.08. While the amounts conversion percentage for this arrangement were in operational condition respectively 41.6, 48.78, 50.96. The results are unexpected. The computational volumes for the operational conversion percentage are respectively 542,610 and 710ml.

The results shown are shown in **Fig. 1**.

Calculated volume for the same percentage of expected conversions are respectively 1000, 1042 and 1110ml. It seems that, they do not differ much from operational volumes.

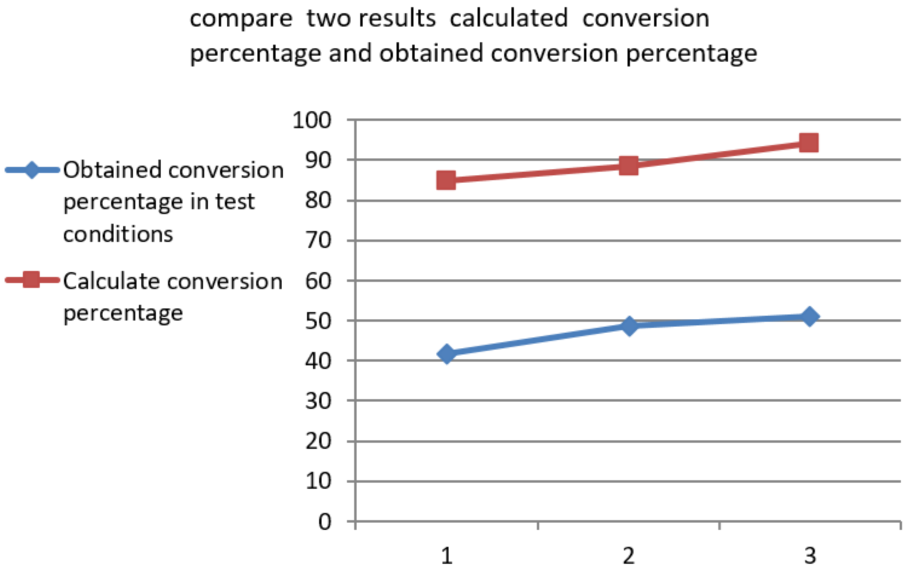

Fig. 1. Compare of results calculated conversion percentage and obtained conversion percentage

CONCLUSION

According to the obtained results in the production of sodium –meta borate by continuous methods and laboratory conditions with two modes:

1) serial reactors with conversion percentage respectively 41.6, 48.78 and 50.96
2) reactors by higher volume with values respectively 1000,1042 and 1110ml

and those are with discontinuous method, it can conclude that if sodium per-borate is considered as the final product and sodium-meta borate is considered the product in first stage, regardless of the second stage because this compound reacting by hydrogen peroxide and no calculation of second stage time. But given the fact that the production operation are in a batch condition have been needed time include filling reactor time, reaction time, cleaning time, empty time, continuous method has been appropriated for saving time. It is good idea to produce sodium-meta borate in continuous conditions but in this study it was found that the production speed is higher in continuous conditions. However, a decrease in the conversion of sodium-meta borate was observed in laboratory conditions, which requires more work in this field and in the future.

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