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2012**

*Technopreneurship Based on Business and Technology
March 23-24, 2012*

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Studying Synthesis Reaction Kinetics
 Of 2,5 Bis (4-Hydroxy-3-Metoksibenzilidin) Cyclopentanone
 From Vanillin And Cyclopentanone in comparison cyclopentanone :
 vanillin, 1:10.

Imam Santosa

Departement of Chemical Engineering, Ahmad Dahlan University

Abstract

The 2,5-bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone or pentagamanuvon (PGV-O) compound is one of the modified structure of middle chains of curcumin compound in which the methyl acetone was replaced with cyclopentanone. Its PGV-O activity as an antioxidant and anticancer in Indonesia. To develop a PGV-O synthesis into an industrial scale, it is necessary to find the reaction kinetics and the influential variables. This research aims to study the kinetics of vanillin reaction with cyclopentanone to yield 2,5 bis(4-hydroxy-3metoksibenzilidin) cyclopentanone (PGV-O) which is run in batch by making a model reaction. Vanillin weighing 22.98 grams (0.15 mol) was added to cyclopentanone as much as 132.6 ml (1.5 mol). The mixture was stirred at 400 rpm. The reaction speed by addition of HCl as a catalyst as much as 0.15 mol. On each interval of 10 minutes, a few mg of sample was taken to be analyzed by spectrophotometry of which the concentration of PGV-O are known. The variables of this study are time (0-60 minutes) and temperature (30-60°C). Data analysis techniques was using the method of average error, square r, and SSE. With the reaction kinetics model which follows the shrinking core model (SCM), at minute 0 to 60 with the stirring, the diffusion through the coating film was concluded, so that the appropriate equation is:

$$\frac{t}{\tau_1} = \ln(1 - X_B)^{-0.5}$$

Key words: kinetics, SCM, PGV-O

1. Problem Background

Exploration of curcumin compounds through chemical synthesis was carried out in 1964 by Pabon using a derivative benzaldehyd (vanillin) and ketone derivatives acetyl aseton complex/B2O3) as starting materials. The reaction was run at room temperature. Further development of Pabon method leads to the thought to produce a synthesis that is much cheaper, simpler and produces a larger number of curcumin compounds and curcumin-like compounds with certain variations in order to obtain an analog of curcumin that is expected to be more stable with comparable activity or more potent, safe, quick and cheap.

In 1960 Kodak synthesized the compound of 2,5-bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone or pentagamanuvon (PGV-O) by means of vanillin condensation with cyclopentanone under acidic conditions used in the mixture of film forming photosensitive polymer making. Its activity as an antioxidant and anti-inflammatory (Deli, 1998) is better than curcumin, So that PGV-O is potential to be developed as a new anti-inflammatory and anti-cancer in Indonesia. Curcumin analogues, 2,5 bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone can be prepared by condensation reaction between vanillin and cyclopentanone for 2 days with a stoichiometri ratio of 2:1 with one part of concentrated hydrochloric acid catalyst to the pressure and room

temperature (Sardjiman, 2000).

This study was purposed to know the reaction kinetics of the synthesis of 2,5 bis(4-hydroxy-3-metoksibenzilidin) cyclopentanone prepared by condensation reaction of vanillin and cyclopentanone with excess cyclopentanone at several temperatures.

B. Research Objectives

This research aims to study the kinetics of vanillin reaction with excess cyclopentanone generating 2,5 bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone which is run in batch, by making a model reaction. The variables studied were time and temperature.

C. Review references

The compound of 2,5-bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone or pentagamanuvon (PGV-O) is one modified structure compound of the middle chains of curcumin in which the acetyl acetone group was replaced with cyclopentanone (Sarjiman, 1994).

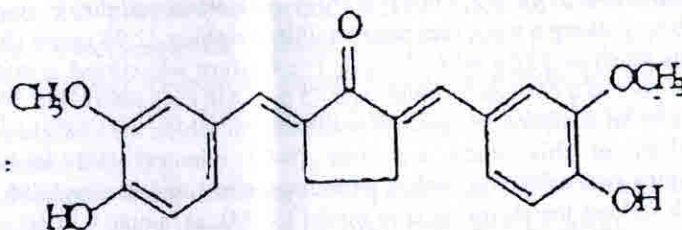


Fig 1. Structure of 2,5-bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone.

Biological activity of these compounds are similar to curcumin, it has been studied the activity of antioxidant, antifungal, antibacterial, and inhibition of sikloosigenase and anti-inflammatory (Sardjiman, 2000). Its activity as an anti-inflammatory (Da'i, 1998) is better than curcumin.

Curcumin analogues, 2,5 bis (4-hydroxy-3-metoksibenzilidin) cyclopentanone can be prepared by condensation reaction between vanillin and cyclopentanone for 2 days with a stoichiometry ratio of 2:1 with one part of concentrated hydrochloric acid catalyst to the pressure and room temperature (Sardjiman, 2000).

There may be two processes of condensation reaction between vanillin and cyclopentanone, namely

1. Cyclopentanone reaction as nucleophiles with vanillin as the electrophile in the acid condition forms PGV-O.

2. Self reaction among cyclopentanones in acidic conditions forms aldol.

Self-forming reaction among cyclopentanones forms aldol compounds was suspected as the equilibrium reaction which the equilibrium tends toward carbon than toward the aldol (Sykes, 1989), so that the second reaction can be ignored.

Nugroho (1998) conducted a study of temperature variations on the mixing process in the synthesis of PGV-O and found the variation of temperature 28°C, 38 ° c, 48°C in stirring obtained were 62.28%, 62.06% and 60.46%. This suggests that the effect of temperature variations on the mixing phase in of PGV-O synthesis was not much different. Husen (1998) examined the variation of vanillin and cyclopentanone problem in the synthesis process of PGV-O, and concluded that a variation of

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cyclopentanone 2:1; 2:1,5; 2:2 obtained were 68.15%, 75%, 71, 2%. The difference in rendement was caused by different amounts of compounds lost during the purification process. Suryanto (1998) examined the research on wait-time to the results yield in synthesis of PGV-O, and summarized the results obtained/yield at optimum wait-time of 6 days. Supriyanto (1999) stated in his research that the results obtained were 58%, 61.7%, 62.5% for each addition of 6 drops, 12 drops and 18 drops of concentrated hydrochloric acid catalyst. Setiawan (1999) stated the technical vanillin as raw materials for the PGV-O synthesis, rendement result was fewer. Kurniawati (1999) examined the methods of quantitative analysis of PGV-O and heksagamanovon-O by spectrophotometry. The best determination for the PGV-O was with 0.1 N NaOH solvent with molar absorptivity of $5.903 \times 10^4 \text{ cm}^{-1}$ and LOD $0.421 \mu\text{m}$ compared to solvent of ethyl acetate and alcohol.

PGV-O synthesis reaction of cyclopentanone and vanillin is a reaction between the solid and liquid in batches, so it is necessary to find information about a reaction like this. Solid granular reaction with liquid which is not or little soluble in the liquid has been investigated by some previous researchers .

Yoshioka et al., (1998) studied the kinetics of hydrolysis of powdered polyethylene terephthalate (PET) with nitric acid catalyst with a modified shrinking core model. PET from waste bottles was degraded at atmospheric pressure in 7-13 M Nitric Acid at a temperature of 70-100°C for 72 hours produced terephthalic acid and ethylene glycol. The kinetics of hydrolysis of PET can be explained by the shrinking core model modification with the controlling chemical reactions of which the effective surface area was proportional to the degree of PET that has not reacted. Reaction rate was proportional to the concentration of nitric acid and inversely proportional to the initial particle size of PET. Reaction activation energy was 101.3 kJ / mol.

Mosig et al., (1997) studied the kinetics and the characteristics of polysuccinimide hydrolysis termal. Polysuccinimide hydrolysis kinetics was studied in the temperature ranged 31-72°C and pH 8 to 10.5. At higher temperatures and lower pH in accordance with the shrinking core model, the chemical reaction controls the overall reaction rate, where the reaction has one orde toward hydroxyl concentration. Reaction activation energy was 35 kJ / mol.

Pavel Šlemín, Gerhard Heide, Aleš Helebrant, (2008) in *Complex Study of E-Glass Corrosion*, examined the kinetics of corrosion of the glass forming SiO₂ fibers using Shrinking Core Models kinetics.

Agnieszka S., Michał I., Zygmunt S., (2006) applied the Shrinking Core Models on the bioleaching of black shale particles to study the possible reaction mechanisms that occur.

Framework of thinking

The reaction that occurs between solid vanillin (B) with a liquid cyclopentanone (A) into PGV-O (C) and water (D) under acidic conditions are:



Acid catalyst affects the reaction rate constants. Steps of the reaction between liquid cyclopentanone with solid vanillin, was approached by the shrinking-core model that can be decomposed into three steps (Levenspiel, 1999):

1. A liquid diffusion through the liquid film around the surface of solids
 2. A substance diffusion through a layer of solid product
 3. Surface reaction between the A liquid with compact solid.
- Equations will be developed to a of solid cylindrical granules reaction conversion form to find which steps control the reaction. The assumption is:

1. Vanillin particles considered in cylindrical solid with a ratio of length (L) of the radius is large enough, and the length of vanillin during the reaction is considered fixed, so the depreciation amount of vanillin is equivalent to the depreciation of its radius.
2. Fixed number of particles during the reaction.
3. reaction was assumed to have the same direction.
4. Isothermal process.
5. When the stirring process, the liquid-solid mixture is considered homogeneous.
6. Fixed acid concentration during the reaction.

Here are the estimated models which can be used to explain the events of the above reactions.

Model I: diffusion through the fluid control film

This model was developed with the assumption :

1. At the beginning of the reaction, there were a lot of cyclopentanone liquid (A), and there were little reaction product, so the diffusion of liquids A to solid surface B through a layer of film is slower than through the cake. The concentration of A on the surface of the cake (exterior) is equal to the concentration of A at the surface of solids (center).
2. The rate of reaction is faster than the diffusivity of A to dense surface of B through the layer of the film.

Based on equation (1) each mole of A reacts with 2 moles of B. Thus the relationship can be expressed by the equation:

$$-2r_A = -r_B \dots\dots\dots(2).$$

The balance sheet for vanillin (B) on a single particle with the assumption stoichiometri reaction conditions:

$$-r_B = \frac{-dN_B}{S_{ex}.dt} = \frac{-dN_B}{2\pi R(R+L).dt} = \frac{-2dN_A}{2\pi R(R+L).dt} = 2k_l(C_{A1} - C_{As}) \dots\dots\dots(3),$$

where: r_B = reaction rate of vanillin,
 S_{ex} = initial surface area of vanillin, m^2 ,
 dN_B = change in concentration of vanillin,
 dN_A = change in concentration of Cyclopentanone,
 dt = change in time,

k_l = mass transfer coefficient between fluid and particle, m/s,
 C_{A1} = concentration of A (cyclopentanone) in the liquid phase, mol/m^3 ,
 C_{As} = concentration of A (cyclopentanone) in the solid phase, mol/m^3 .

Concentration value of A on the surface of B (C_{As}) is very small, so it is considered equal to zero. Concentration of A change in stoichiometri toward B.

$$C_{A1} = C_{A0}(1 - X_A) \dots\dots\dots(4),$$

with: C_{A0} = initial concentration of cyclopentanone,

X_A = reaction conversion of cyclopentanone.
 Substituting the equation (3) and (4) yields:

$$\frac{-dN_B}{2\pi R(R+L).dt} = 2k_1C_{A0}(1-X_A) \dots \dots \dots (5).$$

Conversion of the above reaction can be expressed in the form:
 $1-X_A = 1-X_B$ = volume of solids has not reacted/total volume of particles

$$1-X_A = 1-X_B = \frac{\rho r_c^2 L}{\rho R^2 L} = \left(\frac{r_c}{R}\right)^2 \dots \dots \dots (6).$$

Moles of vanillin in vanillin particle are:

$$N_B = \rho_B V \dots \dots \dots (7),$$

where ρ_B is the density of the solid molar and V is the volume of solid particles. Shrinkage of the volume or the radius of the unreacted solid granules is proportional to the reduction of solid reactant moles.

$$-2dN_A = -dN_B = -\rho_B dV = \rho_B d(\pi r_c^2 L) = -2\pi \rho_B L r_c dr_c \dots \dots \dots (8).$$

With r_c is the radius of the surface toward central cylinder vanillin. Substituting the equation (5) and (8) yields:

$$\frac{-\rho_B L R dr_c}{(R+L)r_c dt} = 2k_1 C_{A0} \dots \dots \dots (9).$$

Integration of equation (9) and rearrangement yields:

$$t_1 = \frac{\rho_B L R}{2(L+R)k_1 C_{A0}} \ln\left(\frac{R}{r_c}\right) \dots \dots \dots (10).$$

Where t_1 is the process time during the diffusion through the controlled film layer. Substituting the equation (6) and (10) with rearrangement yields:

$$t_1 = \tau_1 \ln(1-X_B)^{-0.5} \dots \dots \dots (11), \text{ With}$$

$$\tau_1 = \frac{\rho_B L R}{2(L+R)k_1 C_{A0}} \dots \dots \dots (12).$$

If the film diffusion controls the reaction rate, so the relation of time (t) to mole fraction of solid $\ln(1-X_B)^{-0.5}$ is linear. The value of k_1 can be calculated from the slope constant of linear equations.

Model 2: controlling chemical reactions

These models have been prepared with assumption:

1. In the initial reaction, there were still many cyclopentanone (A), there were a bit reaction product a bit, so that the diffusivity of fluid through the ash layer was quick.
2. Reaction rate is slower than the diffusivity of A to the surface of B through the film layer due to the phase difference.

Changes in cross-section of solid vanillin when the chemical reaction is the

determining step can be seen in figure 3.

Reaction rate is proportional to the solid surface area that has not reacted and is not influenced by the cake.

$$\frac{-dN_B}{2\pi r_c(r_c + L).dt} = \frac{-2dN_A}{2\pi r_c(r_c + L).dt} = 2k_r C_{A1} = 2k_r C_{A0}(1 - X_A) \dots \dots \dots (13),$$

where k_r is the reaction rate constants with one orde for the surface reaction.

Substituting the equation (8) and (13) yields:

$$\frac{-\rho_B L R^2 .dr_c}{(L + r_c)r_c .dt} = 2k_r C_{A0} \dots \dots \dots (14).$$

Assuming $L/(L + r_c)$ equals one, the rearrangement of equation is:

$$-\frac{\rho_B .dr_c}{r_c^2} = 2k_r C_{A0} .dt \dots \dots \dots (15).$$

Integration of equation (14) and rearrangement yields:

$$t_r = \frac{\rho_B .R}{2k_r C_{A0}} \left(\frac{R}{r_c} - 1 \right) \dots \dots \dots (16).$$

Where t_r is the time during controlling chemical reaction. Substituting the equation (16) with equation (6) yields:

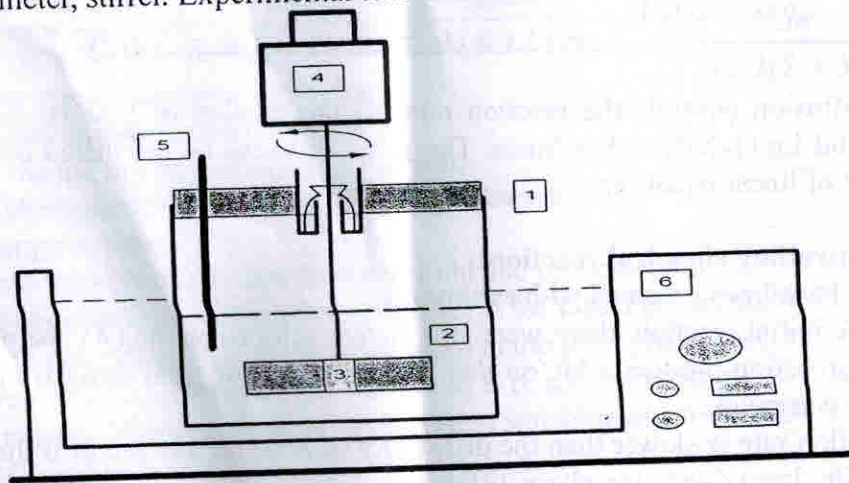
$$t_r = \tau_r [(1 - X_B)^{-0.5} - 1] \dots \dots \dots (17), \text{ with}$$

$$\tau_r = \frac{\rho_B .R}{2k_r C_{A0}} \dots \dots \dots (18).$$

If chemical reaction controls the reaction speed, the connection of time (t) to $[(1 - X_B)^{-0.5} - 1]$ is linear. K_r values can be calculated from the slope of the linear equation constants.

D. How to Research

The tools used in this study consisted of a beaker glass, water bath, thermometer, stirrer. Experimental tool circuit showed in Figure 2.



Remarks:

1. Tank with a cover
2. Solution
3. Mixer
4. Motor Drive
5. Thermometer

Figure 2. The series of experimental tools

Water bath is turned on and set at the reaction temperature. Vanillin weight (0.15 mol) is put into a glass beaker, followed by cyclopentanone 132.6 ml (1.5mol) Then stirred with a speed of 400 rpm, after the mixture homogeneous, HCl as a catalyst as much as 0.15 mol is put in. The temperature is maintained with a water bath, each interval of 10 minutes milligrams of sample was taken and was diluted with 0.1 N NaOH in order to be analyzed by spectrophotometry). The variables studied were temperature (30, 40, 50, 60, 70 ° C) and time in the range 0 to 100 minutes. PGV-O levels formed were analyzed by means of a spectrophotometer. To obtain the concentration of PGV-O, the values of sample absorbance are plotted into a curve. Standard curve was obtained by making the related curve between concentration of PGV-O which is diluted with 0.1 N NaOH toward the absorbance. The observations were at a wavelength of 517 nm.

E. Research Result and Discussion.

In the reaction with vanillin and cyclopentanone with a ratio of 1:10, the relationship between reaction time and conversion toward cyclopentanone is seen in figure 3. An approach to the best value of R² by using the quadratic equation, except at a temperature of 70 C which is more appropriate to use the linear equations because reaction phase was on the on the disappearance of PGV-0 per condition.

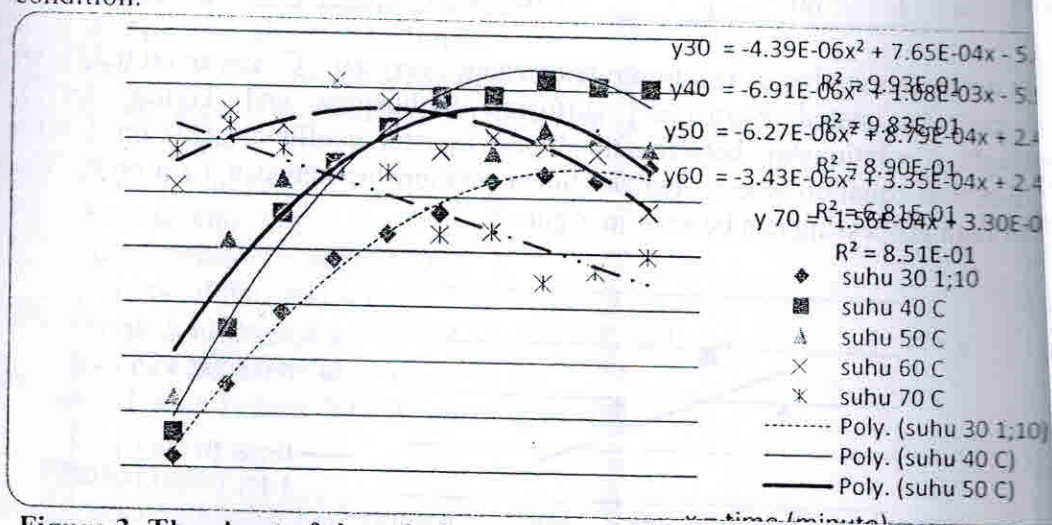


Figure 3. The chart of the relationship between time (minutes) on conversion various temperatures in comparison cyclopentanone: 1:10 vanillin

Kinetic model used as the basis of computation was linear models, so it was suitable if all the experimental data used. In the analysis of kinetic models, it was selected 5 initial data from 0 to 50 minutes and the temperature range 30 C -

C. The data showing the disappearance of PGV-0 products were not analyzed.

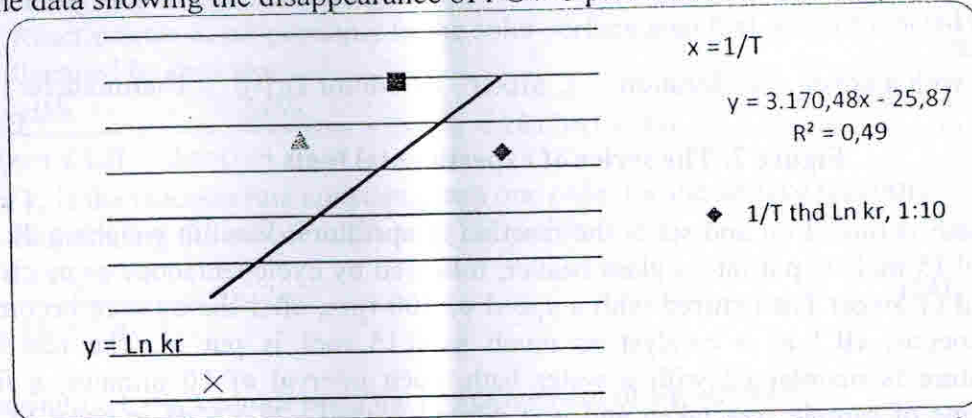


Figure 4. The relationship between $\ln kr$ to $1 / T$ in Model 2 in comparison cyclopentanone: 1:10 vanillin.

It appears from Figure 4. that the relationship between the straight line curve data points is not good enough. This is because phase reactants are not homogeneous. The relationship between kr with T follows the Arrhenius equation with the equation:

$$k_r = -25,87 e^{3170,48/T} \dots\dots\dots(19)$$

Table 1. The increase in the value of each reaction rate constant at 10°C in comparison cyclopentanone: 1:10 vanillin.

No.	Temp., $^\circ\text{C}$	Math value of k_r , m/s	k_{n+1}/k_n
1	30	2.04E-07	0.715838
2	40	1.46E-07	0.73081
3	50	1.07E-07	0.744705
4	60	7.94E-08	0

The increase in value of reaction rate constant every 10°C was about 0.525, so that it can be stated controlling diffusion (Johnstone and Thring, 1957). Then sought a relationship between the liquid transfer coefficient (kl) on T with Stokes-Einstein equation where obtained linear relationship between T / μ on kl . The results of data processing can be seen in Figure 5.

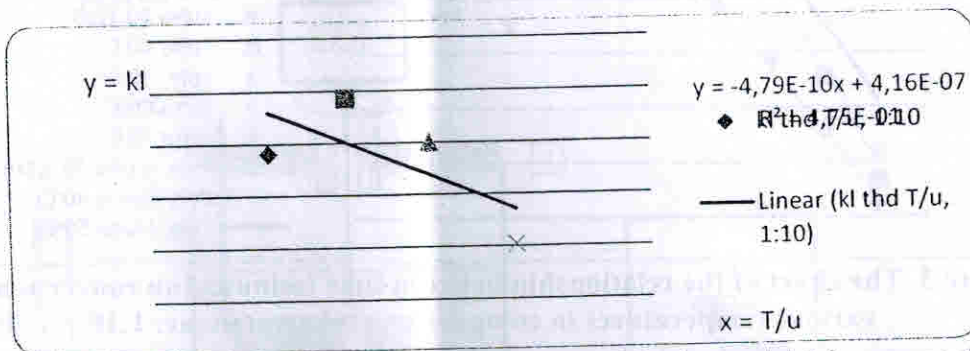


Figure 5. The relationship between kl against T / μ in model 1 in comparison cyclopentanone: 1:10 vanillin.

The relationship between the liquid transfer coefficient (kl) on T is:

$$4.79 \cdot 10^{-10} \text{ kl} = T / \mu + 4,16 \cdot 10^{-7} \text{ (m / sec)} \dots\dots\dots (40).$$

As a comparison value for KOH = $4,3 \cdot 10^{-6} \text{ m / s}$ are $10^{-6} \text{ NaOH} = 1.15 \text{ m / s}$.
(Levenspiel, 1993).

E. List of Symbol

- A = frequency factor, cm/min
 B = baffle width, cm
 C_{AC} = concentration of A (cyclopentanone) on the surface of solid vanillin, mol/m³
 C_A = concentration of A (cyclopentanone), mol/m³
 C_{AL} = concentration of A (cyclopentanone) in the liquid phase, mol/m³
 C_{AS} = concentration of A (cyclopentanone) in the solid phase, mol/m³
 D = diameter of stirrer/mixer, cm
 D_t = diameter of tank, cm
 D_e = effective diffusivity coefficient of liquid reactant in the product layer, m²/s
 E = activator energy, cal/gmol
 k_r = reaction velocity constant, m/s
 k_f = mass transfer coefficient between fluid and particle, m/s
 L = average length of the early vanillin solids, m
 N = speed of stirring, rpm
 R = initial radius average of the vanillin, m
 r = radius of the solid products toward central cylinder of vanillin, m
 r_A = rate of reaction of cyclopentanone
 r_B = rate of reaction of vanillin
 r_c = radius of the surface toward central cylinder of vanillin, m
 v_B = molar volume of vanillin, mol/m³
 S_c = surface area of solid vanillin, m²
 S_{c0} = initial vanillin surface area, m²
 S_r = surface area of a cylinder with radius r, m²
 t = time, minutes
 τ = constant, minutes
 T = absolute temperature, K
 V_{ii} = Cyclopentanone volume, ml
 V_t = Volume of the tank, ml
 XB = mole fraction of vanillin that has reacted, mol/mol
 μ = viscosity of cyclopentanone, g/cm sec
 s = the advance voltage between the phases, dyne / cm

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