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RELIABILITY OF FLOW INJECTION SYNTHESIS FOR THE CUZN FERRITE FORMATION

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Abstract

Reliability Instrumentation is an absolute requirement that must-have instrument, namely the behavior of the reaction are reliable and valid with basis both Avrami, Ozawa and Kissinger. The equation plus cooling rate must be in Multiple Regression Corelation, obtained based on the classification Gaiford, where High reliability values signaled where the instrumentation system formation history of the formation of materials with the principles of reliability Avrami has a relatively high and not allways for Ozawa . All sample react and data notice by Flow Injection Synthesis-FIS. From previously as pure alkaline untill all raw material be yield mixing with akaline exess. The raw material mixing must be design in relatively precise, and in exess alkaline in norder to easy to disposible and continued process. In this researchs we try to processe of the data of yield of $Cu_{(1-x).}Zn_x$ Fe_2O_4 in six diversification. The result of the process in multiple corelation give reliable and valid.

Keywords: Reliability, reliable, valid, Avrami, Ozawa, Kissinger, cooling rate, Flow Injection Synthesis, diversivication

1 INTRODUCTION

Flow Injection Synthesis occurs when the acidic salt material in this case drop wise of mixing; FeCl2.4H2O, FeCl3.6H2O ,CuCl2.2H2O,and ZnCl2 , to reacted with NaOH (alkaline) liquid material, forming sediment yield Cu (1-x) ZnxFe3 O4 followed by heat. The reaction process conducted drop wise called Flow Injection Synthesis (FIS). The reaction process will follow the rules of Avrami, Ozawa and Kissinger. If following the determination process Determinants Avrami very high, very low Determinants Ozawa, Kissinger determinant = 1 then it is difficult to determine whether the process Avrami Ozawa including acceptable or not. But because the process Avrami Ozawa is interrelated processes then the calculation must be related namely to multiple corelasi, then the calculation must follow multiple corelasi process, where determinant must follow Spearment Brown $r_{11} = \frac{2.R}{1+R}$. [1-3] 1

The Avrami equation is $Y(t)=1-e^{-(k.t^n)}$ where k and n as kinetik reaction constan.n as Avrami exponent to express nucleation mechanism. Ozawa equation is $1-Y(t)=Exp\{-K(t)/|a|^m\}$, Y(t) as crystallization degree, K(t) Crystall rate formation, α cooling rate crystall formation,m Ozawa exponent depent on demention of crystall formation. Cooling rate expression by a where decreasing process temperature at any time absolut and Kissinger equation as $d\,Ln(I\alpha I/T^2)/d(1/T)=-(\Delta E)/R$.

The synthesis of uniform-size or mono disperse, with a relative standard deviation of < 5% nano crystals in the physics characteristic of nano material such as CuZnFerrite is key importance because the properties of these nano crystals depend strongly on their dimensions [4].Concerning of the Non-isothermal reaction need two fundamental equation mainly Avrami and Ozawa model. Avrami model as shown in equation : Log(-ln(1-Y(t))=n.Log(t) + ln K(T). n determine Avrami exponent, K(T) is the crystallization rate constant where Log k(T)/λ=log K_c. $t_{1/2}=(\ln 2/K_c)^{1/n}$,to determine Avrami exponent (n) we need the plot of ln(-ln(1-X(T)) against lnt . Ozawa model as equation Log(-Ln(1-Y(t)=m Log(α) Both formation parameter were builds, then execute depend on the process system. There are many equation s and parameters have parameters of the dependences to execute until the equations have numerical value reliable or not and we chance the Avrami Ozawa couples that have reliable, but once of pairs may gives high reliability.

I.1 Estimation of Activation Energy Crystals Formation Cu Zn Ferrite.

The thermodynamic test is a quantitative test that involves a certain amount of energy involved in the formation of the material. Therefore, the data required pH, temperature T (K) and time (t) which recorded by the data logger in each of each sample experiments. The data obtained pH, temperature T (K) and time (t) for samples with label code bath02, bath04, bath06, bath078, bath088, and bath09 annexed to this desertation. From the data on each sample estimates can be made provisions the Avrami graph.

Table 1.1 Raw Materials stoichiometry To Yield 0.06 Mol Cu-Zn Ferrit

	$ZnCl_2$	$ZnCl_2$	$CuCl_2.2H_2O$	$CuCl_2.2H_2O$	$FeCl_2.4H_20$	$FeCl_2.4H_20$	$FeCl_3.6H_2O$	$FeCl_3.6H_2O$	Na(0H+	Na(0H+
Bath 04P	0.024	3.27	0.036	6.13	0.04	7.95	0.08	21.63	0.44	17.6
Bath 06P	0.036	4.90	0.024	4.09	0.04	7.95	0.08	21.63	0.44	17.6
Bath078P	0.047	6.38	0.013	2.25	0.04	7.95	0.08	21.63	0.44	17.6
Bath088P	0.053	7.20	0.007	1.22	0.04	7.95	0.08	21.63	0.44	17.6

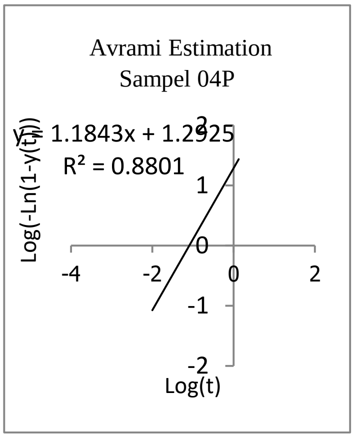
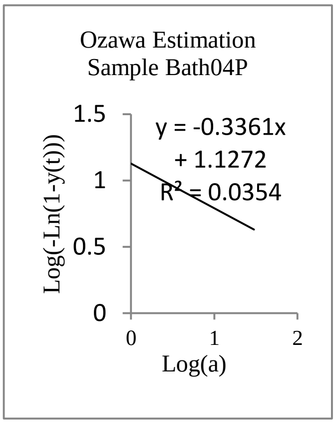
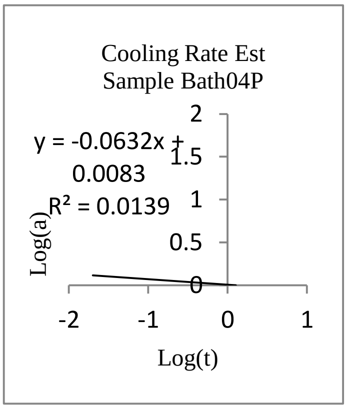
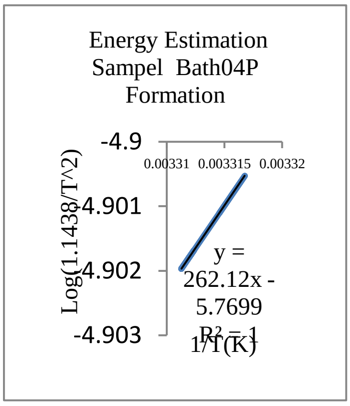
Stoichiometric amount of mass in [g] of each of each salt chloride dissolved in 20 milli liter aqua DM so that the alkaline pH of about 1. While 17.6 grams of NaOH dissolved in 20 milli liter Aqua DM. To condition the yield in an atmosphere of an alkaline pH of around approximately 13 20ml NaOH solution was added to 880 ml of pure H2O to pH = 14 - (- LOG ((17.6 / 40) /0.88)) = 13.69. Number of 880 ml capacity is also a critical reactors, If the volume of saline solution plus 80 ml to 960 ml volume of the solution will meet the capacity of the reactor 1 Liter. In the same way acquired a table similar , for sample bath06, bath078,and bath088 samples.

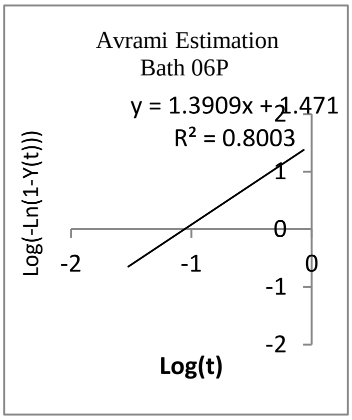
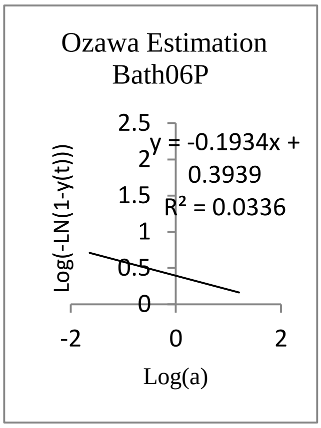
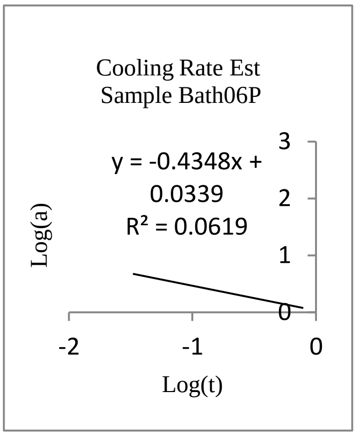
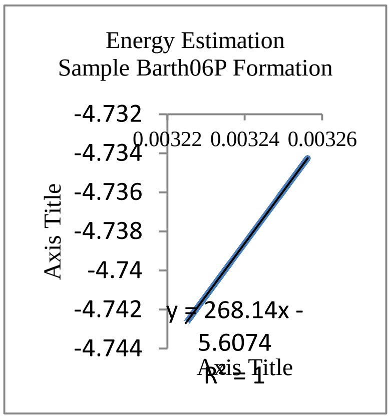
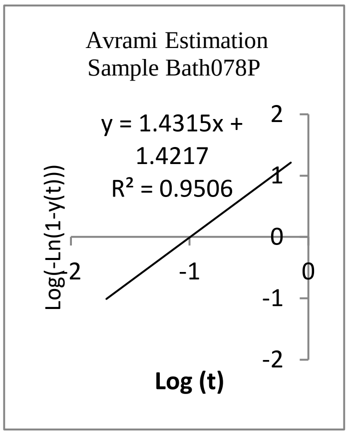
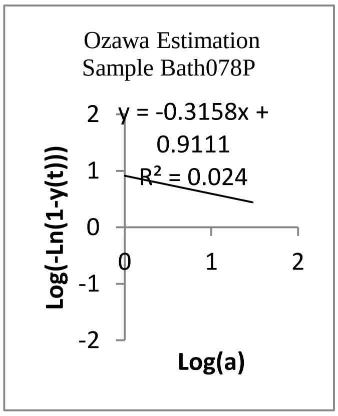
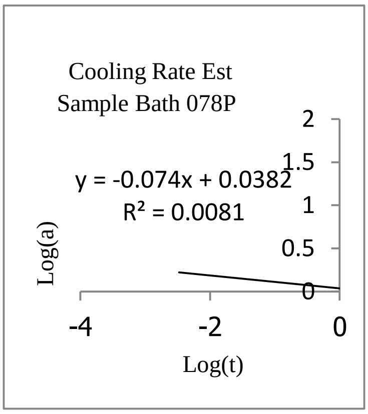
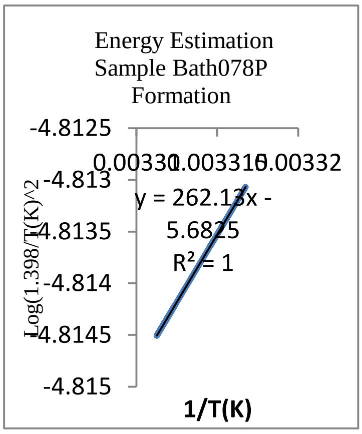
The linier of graph; Avrami, Ozawa, Cooling Rate and Forming Energy of Bath 04P. In other words, the equation that appears in the Graph Logarithmic Linear Estimation Process Formation Energy of the material Bath04P CuZnFerrite with sample code, 06P, 078P, and 088P tabulated in Table 2.1, Applications Avrami-Ozawa equation Kissinger results are represented in Table 2.1 so that the final result in the form of the estimated energy yield formation ZnCuFerrite material particular. Retrieved estimated average energy yield formation ZnCuFerrite is 2190 [joules / mol].

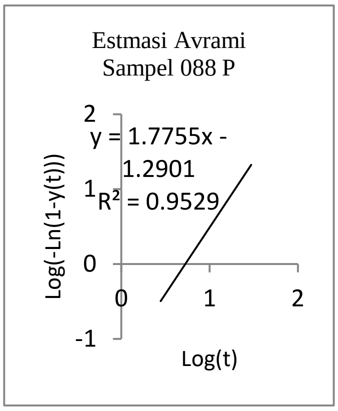
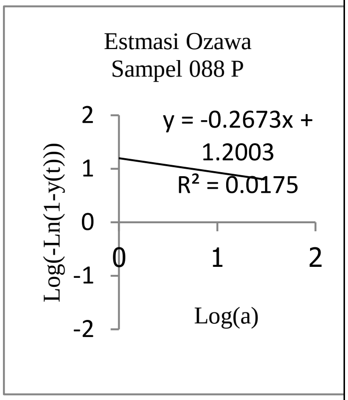
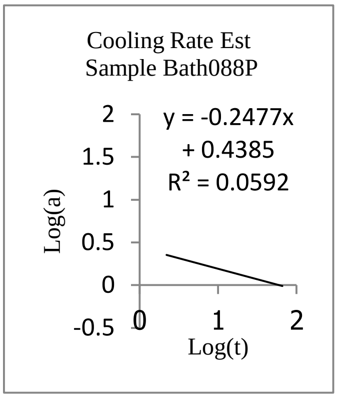
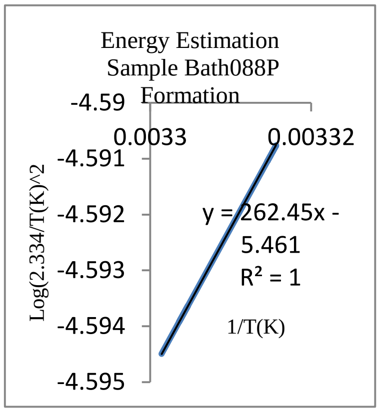
Table 1.2..Review of All Linear Equation Avrami-Ozawa-Kissinger (Bath04P to Bath09P)

Sampel	Arami	Ozawa	Cooling Rate	Kissinger
Bath04	y=1,1843 X+1,2925	y=-0,3361 X+1,127	y= -0,0632X+0,008	Y= 262,13X-5,682
Bath06	y=1.3909 X+ 1.471	y=-0.1934 X +0.394	y=-0.4348 X + 0.034	Y=267.67X-5.607
Bath078	y=1.4315X+1.4217	y=-0.3158 X +0.911	y=-0.074X+0.911	Y=262.13X-5.682
Bath088	y=1,776X-1,2901	y= -0,2673X+1,200	Y =- 0,2477X+0,438	Y=262,45X-5,461

Table 2.1 Linier Logaritmic graph of the energy forming Process of CuZnFerrite materials of sample code Bath04P,06P,078P,088P.

<table>
<tr><td colspan="4">Table 2.1. load Ozawa Avrami parameter for samples and related bath04P.
the determination of the constant cooling rate and the estimated energy of formation of the sample material.
In detail all linear equations described in Table 2.1</td></tr>
<tr><td>Avrami Parameter</td><td>Ozawa Parameter</td><td>Cooling rate Parameter</td><td>Material Formation Energy</td></tr>
<tr><td>
</td><td>
</td><td>
</td><td>
</td></tr>
<tr><td>y=1.184x + 1.292
R^2 = 0.88</td><td>Y=-0.336x + 1.127
R^2=0.035</td><td>Y=-0.063x + 0.008
R^2 =0.013</td><td>Y=262.1x-5.769
R^2=1</td></tr>
<tr><td colspan="4">Table 2.2. load Ozawa Avrami parameter for samples and related bath06P.
the determination of the constant cooling rate and the estimated energy of formation of the sample material.
In detail all linear equations described in Table 2.2.</td></tr>
<tr><td>Avrami Parameter</td><td>Ozawa Parameter</td><td>Cooling rate Parameter</td><td>Material Formation Energy</td></tr>
</table>

			
Y=-1.390x+1.471 R²=0.800	Y=-0.193x + 0.393 R²=0.033	Y=-0.434+0.033 R²=0.061	Y=268.1x -5.607 R²=1
Table 2.3. load Ozawa Avrami parameter for samples and related bath078P, the determination of the constant cooling rate and the estimated energy of formation of the sample material. In detail all linear equations described in Table 2.3			
Avrami Parameter	Ozawa Parameter	Cooling rate Parameter	Material Formation Energy
			
Y=1.431x + 1.421 R²=0.960	Y=-0.315x + 0.911 R²=0.024	Y=-0.074x + 0.038 R²=0.008	Y=262.1x – 5.682 R²= 1
Table 2.4. contains Ozawa Avrami parameter for samples and related bath088P the determination of the constant cooling rate and the estimated energy of formation of the sample material. In detail all linear equations described in Table 2.4			
Avrami Parameter	Ozawa Parameter	Cooling rate Parameter	Material Formation Energy

			
Y=1.775x – 1.290 R^2=0.962	Y=-0.267x + 1.200 R^2=0.017	Y=-0.247x + 0.436 R^2=0.059	Y=262.4x – 5.461 R^2=1

1.2. The continued discussion of parameters associated with processing parameters

The forming material particles, such as; Avrami parameter, parameter Ozwa and parameters related to the energy crystals forming material. In the Avrami parameter equation y stated Log (-Ln (1-y (t))) and x expressed Log declare t with an intercept and slope k Log declared parameter n in the equation y as eqution Ozawa Log (-Ln (1-y (t))) and x expressed Log (a) with an intercept stated Log K (T) as well as a linear equation. Slope declared parameter m. The Parameter a stated temperature changes riel process at any time. On cooling rate equation is expressed by the equation Log (a) = log F (T) -b logt , So Y express Log (a), Intercept equation expressed Log F (t), so that F (t) = 10 ^ (LogF (t)). .[7-10]. With conventional algebra and algebraic calculations can be determined logarithmic, among others; Φ-constant cooling rate and AE - crystal formation energy yield.

1.3 The Crystal formation CuZnFerrite Fisbath04 of the sample code

The Calculation of the Estimated Energy Establishment of Material Ferrite Fisbeth04P .Data pH and temperature samples Bath04P shown in appendix B1. In the table datalogger fisbath04P Settling time = 33 sec, Avrami equation: log [-ln (1-y (t)] = log k + n log t , From the linear graph Avrami a picture 2.1 for the samples obtained Bath04P 1.184X + y = 1.2925 in order to obtain the parameters of Avrami; n = 1184, log k = 1.2925 and k = 19.61. Furthermore Ozawa estimation using the following equationOzawa equation: log [-ln (1-y (t))] = log K (T) -mlog (α), From the graph 4.2.1 b Ozawa estimation equation is Y = -0.3361X + 1.1272, Parameter Ozawa; m = 0.3361 K (T) = 10 ^ 1.1272 = 13.4, Estimated cooling / heating rate Log α = Log F (T) - b log t ie as Y = - 0.0632X +0.0083,b = -0.0632, F (T) = 10 ^ 0.0632 = 1.1566, K (T) / K (c) = F (t) m = (1.1566) 0.3361 = 1,050

α = log k / logKc; K (c) = 13.4 / 1.050 = 12.76 Log K (c) = 1,105, log k = 1.2925, Overall Cooling Rate Φ = 1.2925 /1.105 = 1.169 then the cooling rate = 1,17oC / minute], crystalisasi energy Kissinger method as follows; AE = dY / dx = 262.12 x 8:31 J / mole = 2,178.22 J / mol, = 2,178.22 x 239 x10-6 kcal / mol= 0521 kcal

Applications constants used in the linear equation Φ Kissinger useful to determine the crystal formation

energy material.[7-11] AE was obtained from the slope of the linear graph equations Log IΦI / T2 versus 1 / T. Presentation of calculations carried out in two stages, namely the establishment of linear equations of the datalogger is contained in Table.2.2 . On the table were made in a few lines consisting of; bris sample Bath04P, Bath06P, Bath078P,and Bath088P . Each line is made in four columns where each column contains a linear graph, among others; Log linear graph (-Ln (1-y (t))) versus log (t) to obtain a linear equation of the line that explores the Avrami constants, graphs Log (-Ln (1-y (t))) versus log (a) to get the equation of the line that explores the constant Ozawa, Log graph (a) versus log (t) to get the equation of the line that explores the rate constant changes in temperature and graph process α Log (α / T2 (K) versus log (1 / T) forget the equation of the line that explores the energy of the reaction forming material.
Tabulate equality is incorporated in Table 3 d is referred to as a multiple correlation regression equation that is intended to get constant Avrami, Ozawa constants, and the constant rate of change of temperature - cooling / heating rate Φ.For the purposes of acquisition of multiple correlation koefisisien use table 4. The table is the regression coefficient matrix table 3 X 3 concerning, 3 lines. Each cell contains a determinate constant and the regression coefficients. In the 3X3 matrix, contains a section line P, LT , which P dependent variable Log (-Ln (1-y (t))), LT independent variables Log (t) and LA-free variable Log (a). Some cell is formed; Cell (P, Lt), cells (P, LA) and cells (La, Lt).Cell (P, Lt) containing R 2 and R regression equation Log (-Ln (1-y (t))) vs. Log (t). Cell (P, La) R2 and R contain the regression equation Log (-Ln (1-y (t))) vs. Log (a). Cells (La, Lt) containing R2 and R regression equation Log (-Ln (a)) vs. Log (t).

At the bottom of the table cell contains double correlation value according to the equation3.7 and in accordance with the reliability equation equation 3.6- Spearman Brown. Here are the results listed in table calculation; Reliability r Sample Bath04P = 0.966, r = 0.928 Bath06P Sample, Sample bath0788 r = 0.986, r = 0.987 bath088P Sample, Sample Bath09P r = 0.946.

Table. 3.0 Calculation; Reliability r Sample Bath04P = 0.966, r = 0.928 Bath06P , bath0788 r = 0.986, r = 0.987 bath088P ,

$$R_{y,x1,x2} = = \sqrt{\frac{(r_{yx1}\,2 + r_{yx2}\,2) - (2r_{yx1} x r_{yx2} x r_{x1x2}}{(1 - r_{x1x2}\,2)}}$$

Fung.	Sample 04P			Sample 06P			Sample 078P			Sample 088P			Sample 09P		
	P	**LT**	**LA**	**P**	**LT**	**LA**	**P**	**LT**	**LA**	**P**	**LT**	**LA**	**P**	**LT**	**LA**
	0,881		0,0139	0,803		0,061	0,951		0,008	0,953			0,80		0,017
	0,9			0,8961			0,975		0,09	0,976			0,89		0,128
	0,035			0,0336			0,024			0,059			0,01		
	0,188			0,183			0,155			0,243			0,11		
R	**0,935**			**0,8657**			**0,97**			**0,974**			**0,89**		
R^2(S	**0,874**			**0,7494**			**0,94**			**0,948**			**0,79**		
r	0,966			0,928			0,986			0,987			0,94		

NB :		
	P=Log(-Ln(1-	R (p,lt,la) As Multiple Corelation between P function of LT and LA R(Square)= Regression determinant
	LT=Log(t)	r = Numerical Value of reliability instrument system
	LA=Log(a)	

Bath09P r = 0.946

Based on linear graph listed in Table 3.1 can also be obtained constant determinant coefficient R2, so that the value of the declared value R corelation linear equation is concerned. Parameter R2 accompanying linear equation Avrami, Ozawa, cooling rate are summarized in Table 4.4 to be used as the calculation of multiple correlation coefficients Avrami-Ozawa equation, which theoretically explained in chapter 3.3.2.

2.1 The level of reliability of measurement Instrumentation System FIS-Batch
In the Sample Bath04 Until Bath09 (In Table .2.1 and Table 2.3) were obtained that the correlation, regression determinants and measurement instrumentation reliability values as in the table below;

Table 2.5 Value-R correlation, regression constant determinant-R2, and constant reliability-r. Materials and Systems FIS Instrumentation .

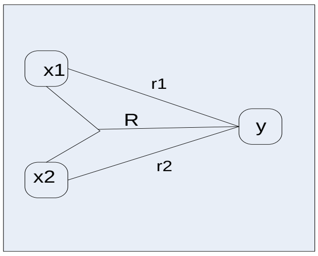
Sampel	Correlation value of regression. -R	The constant value of Regression determinant -R2	Value reliability r
Bath 04P	0,953	0,874	0,966
Bath 06P	0,866	0,749	0,928
Bath 078P	0,973	0,974	0,986
Bath 0.88P	0,974	0,948	0,987

The reliability coefficient obtained based on the classification Gaiford as following : r> 0.8 = very strong, 0.5 <r> 0.8 = strong 0.4 <r> 0.6 = moderate ,So that the provisions constant cooling rate -Φ all samples (from bath 04P and up 06P) such as very good.
In case the provision of the estimates of energy (enthalpy) forming material using Kissinger equation - d (Log IΦI / T2) / d (1 / T) which produce determinant regression R2 = 1 can be qualify for estimating the energy value AE / R where R as a general ideal gas constant = 8.31 J / Mol K. High reliability values signaled that the instrumentation system is seen that the concept of energy estimates CuZnFerrite materials and concept formation history of the formation of materials with the principles of reliability Avrami has a relatively high compared to the concept of material growth with the principles Ozawa. At the quantitative reliability test performed by knowing the level of the coefficient determinant-R2, where R = √ R2 is a correlation coefficient. The level of reliability of research results is a function of the correlation coefficient R can be determined using Spearman Brown equation, with r11 as the value of reliability and R as the correlation coefficient . The coefficient of reliability was good to be in the range of 0.7 (quite good, and 0.8 (good). Correlation Equation Avrami-Ozawa.[15-16].The Linear Equation Avrami-Ozawa may be a correlation, where between one linear equation with other linear equations are still intertwined . The Components are 3 equations , linear correlation doubles as pertaining to the regression equation with correlation coefficient Ryx1, Ryx2 and Rx1x2. Regression formed with U = Log {-Ln (1-y (t))} as a temporary dependent variable, x1 = Log (t), x2 = log (a). independent variables and the dependent variable X1 X2 independent variables.

Multiple correlation coefficient was calculated by the equation

$$R_{y,x1,x2} = \sqrt{\frac{(r_{yx1}{}^2 + r_{yx2}{}^2) - (2.r_{yx1}\ .r_{yx2}{}^2.r_{x1x2}\ )}{(1 - r_{x1x2}{}^2)}} \qquad \text{.................... .3.7}$$

where r yx12, ryx22, rx1x22 (determinant coefficient r square or r2) will be obtained automatically at each program gave rise to a linear equation excel. [12,13]
Information. R2ux1 = Coeff. of Determinant U to x1, R2ux2 = Coeff. of Determinant U to x2, x1 R2x2x1 = Coeff.of Determinant X2 against, Rux1 = Coeff correlation function u to x1, Rux2 = Coeff correlation function u to x2, Rx2x1 = Coeff correlation function x2 against x1 Regression-Correlation Diagrams Estimation of Activation Energy Materials Zinc Ferrite, described as follows;



Regression-Correlation Diagrams Estimation of Activation Energy Materials Zinc Ferrite, described as follows;

Caption circuit
Y (t) = fraction yields equation
Y = equation U = Log {-Ln (1-y (t))}
Y-X1 = Log {-Ln equation (1-y (t))} V / S Log (t)
Y-X2 = Log {-Ln Equation (1-y (t))} V / S Log (t)
X2 X = equation x2 v / s x1
Multiple correlation coefficient R =
r1 = Coef correlation X1 Y-
r2 = Coef correlation Y-X2

From the diagram above can be written where the correlation value R2 component and determinant coefficient value R as the following table;

Table 3.6. Mapping multiple correlation parameter variables Y, X1 and X2 as follows

Variabel dependent	Variabel independent		
	U	**X1**	**X2**
U	**-**	**-**	
X1	**R^2 UX1, R** ux1	**-**	**R^2 X2X1, R x2x1**
X2	**R^2 UX2, R** ux2	**-**	

Rx1x2 double correlation parameters determined and calculated by the equation 3.6, followed by calculation of the reliability parameters r11 by using the equation 3.7. The multiple regression have 5 parameter, mainly; t,Y(t) , a, , Log(-(1-y(t)),,

3.1 Significance Test Validity and Reliability.

The significance level is a tolerance limit to allow the fault of the results of the hypothesis of the value of the population parameter. The level of significance is the probability of getting the price of X in critical areas, if the hypothesis H0 is true. P determination level of significance (α) depending on the level of research is conducted (experiments), if we tested something important or dangerous or urgent, the level of significance to be taken relatively small such as: α = 5%, α = 1%.[13-14]. To determine whether the interpretation of the validity correlation regression significant or not it is necessary to the validity of the testing process that is performed on each parameter equation and the results can be observed through the F-count results were compared with the F-table, where the F-table can be obtained from the Fisher distribution table based on parameters such degrees of freedom df amount of data, the number of regression, which allowed ingkat α error -5%, If F- count> F-table <the acquisition of data is considered valid, where as if the F-table> F-calculate the acquisition of regression are considered invalid .[15-16]

4. Conclusion.

1. CuZnFerrite as multiple corellation have 5 parameter, mainly; t,Y(t) , a=dθ/dt, Log(-(1-y(t)),,Distribution of Fisher classification 1 direction, tail distribution only 1 right end portions of 5% or 1% . Here include as multiple corellation.

2. The articles with yield of bath 04P to 088P**,** was Multiple correlation**.** The articles bath 04P to 09P were reliable and valid article and as Multiple correlation coefficient Reliability R= 0.7. (0.5 <r> 0.8 = strong r> 0.8 = very strong, 0.4 <r> 0.6 = moderate ..

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