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RESEARCH PAPER

Design of reactor for the production of zinc oxide nanoparticles

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Abstract: This study aims to design a continuous stirred tank reactor (CSTR) type reactor to produce zinc oxide (ZnO) nanoparticles. Mass balance calculations were carried out in this study as a benchmark to determine whether the reactor was working properly by knowing the flow of incoming raw materials and the products produced by the reactor. Furthermore, the reactor's design and the stirrer used in the reactor are calculated manually using Microsoft Excel. Based on the calculation results of the reactor design, the reactor volume is 8224.359 liters, with a vessel diameter of 73.298 in, a cylinder height of 166.090 in, and a cylinder thickness of 73.444 in. The top cover of the reactor measures 12.387 inches with a thickness of 0.072 inches, while the bottom cover measures 21.185 inches with a thickness of 0.083 inches, so the overall height of the reactor is 37.552 in. The reactor is equipped with one stirrer with an impeller diameter of 36.722 in, impeller height from the bottom of the tank is 24.433 in, impeller width is 7.344 in, and impeller length is 9.180 in. Turbulent stirring flow conditions with a standard motor power for the stirrer is 6.849 HP. This design will be a reference for building more economical, efficient, and highly demanding reactors.

Keywords: ZnO nanoparticles, reactor design, CSTR, mass balance, reactor stirrer

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INTRODUCTION

Zinc oxide (ZnO) is an n-type semiconductor from group II-VI, with a wide band gap of 3.2 eV at room temperature [1]. This metal oxide has properties that are environmentally friendly, non-toxic, and resistant to corrosion. This material has been widely studied for its activity as antifungal [2], antibacterial [3], anti-inflammatory [4], antimicrobial and antioxidant [5], anticancer [6], sunscreen [7], photoanode in solar cells [8], photocatalyst [9], and anticorrosive [10]. The characteristics and performance of ZnO are highly dependent on particle size, shape, and morphology [11]. The synthesized ZnO particles can produce micro-sized particles into nanoparticles. Based on previous studies, ZnO nanoparticles have been widely used as rubber accelerators [12], efficient adsorbents [13], gas sensors [14-15], and photocatalytic degradation [16].

Several synthesis methods have been carried out to obtain ZnO nanoparticles, such as the direct deposition method [17-18], the homogeneous deposition method [19], the sol-gel technique [20], and the hydrothermal processing method [21-22]. Currently, many industries for manufacturing ZnO nanoparticles use homogeneous precipitation methods. However, a simpler method with cheaper raw materials is needed, namely the direct deposition method. The direct precipitation method has several advantages; it can produce better particle size and morphology, finely dispersed active substances, and easy control of particle size as desired.

Various researchers have proposed several methods to prepare ZnO nanoparticles using direct precipitation methods, such as adding surfactants [23] or using ultrasound fields. Hong *et al*. described how to synthesize ZnO nanoparticles by mono dispersion direct precipitation method using a diameter of 30 nm. $ZnAC_2.2H_2O$ and $(NH_4)_2CO_3$ were slowly dripped into a vigorously stirred polyethylene glycol (PEG) solution, while surface modification of the synthesized ZnO nanoparticles was carried out by capping with oleic acid and SiO_2 [24]. Siqingaowa *et al*. prepared ZnO nanoparticles with $ZnCl_2.2H_2O$ and $(NH_4)_2CO_3$ as raw materials through the direct precipitation method with the help of an ultrasonic field, resulting in an average grain diameter of 12 nm [17].

In the industry, to carry out the synthesis of a chemical, a tool is needed as a place where a reaction occurs, called a reactor. A reactor is a device that acts as a place for a reaction to occur, whether it's a chemical reaction where in this reaction, a material can change from one form to another. In making reactors, it must be ensured that a reaction will produce the highest efficiency toward the desired output product. It is so that the industry that makes the reactor can minimize operational costs to obtain the maximum product. So in this research, to optimize the production of ZnO nanoparticles, it is necessary to design an efficient and accurate reactor design so that it can be useful as a reference in designing reactors and as a teaching and learning method for the design process, working mechanism, to the performance of the reactor.

MATERIALS AND METHODS

Synthesis of ZnO Nanoparticles

The manufacture of ZnO nanoparticles is based on research carried out by previous researchers [25]. The first step that must be done is to dissolve NH_4HCO_3 and $ZnSO_4·7H_2O$ in deionized water. Then the dispersed feed is pressed through the membrane into the microchannel to mix with the continuous feed coming from the continuous feed inlet. The two solutions are mixed in the microchannel, which will lead to saturation of the zinc hydroxy carbonate and produce a crystalline product. After this process, the by-product $(NH_4)_2SO_4$ was removed by washing the powder with distilled water at room temperature (298 K) until no white precipitate was produced when the washing water was tested with a 6% $BaCl_2$ solution. After that, the precursor was washed with ethanol three times. The washed powder was then dried at 100°C overnight, followed by calcination. All precipitation experiments were carried out at room temperature. The schematic and PFD of the ZnO nanoparticle manufacturing process using the direct precipitation method are shown in Figures 1 and 2.

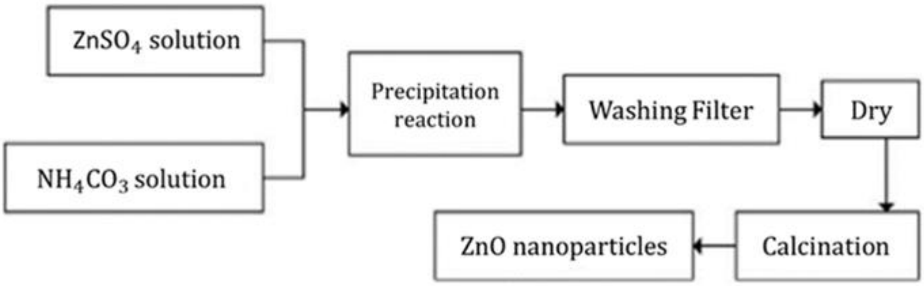

Figure 1. The overall procedure of preparing ZnO nanoparticles

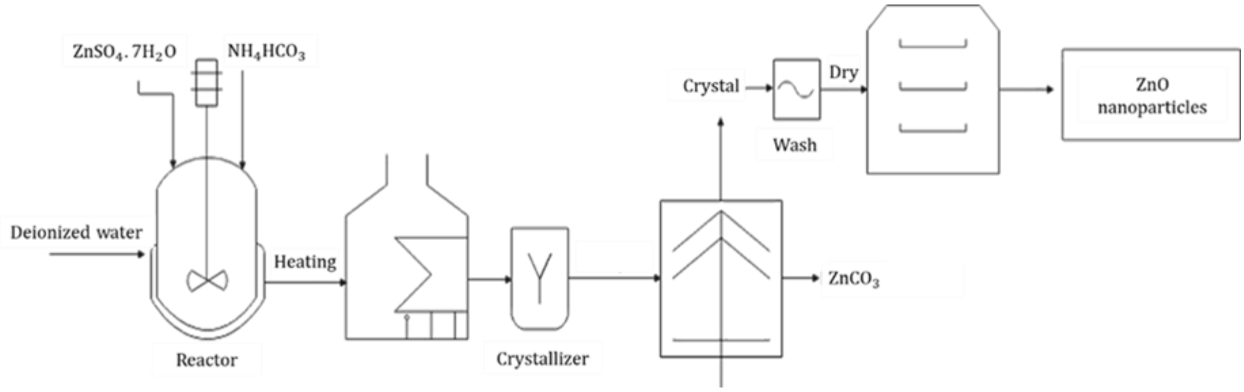

Figure 2. PFD on the manufacture of ZnO nanoparticles

Mathematical Models for Designing Reactors

Table 1 shows the reactor parameters to be calculated. Data analysis is in the form of manual calculations using basic Microsoft Office applications based on equations 1-20.

Table 1. Calculation of reactor parameters

No	Section	Parameter	Equation	Eq
1.	Reactor Planning	Volume Reaktor (V_{total})	V_{total} = V material + V free space	(1)
		Vessel Diameter (Di)	V_{total} = V top lid + V cylin + V top cov $V = \frac{\pi di^3}{24 \tan 1/2\alpha} + \frac{\pi di^2}{4}$ x Ls + 0,0847 di^3 Di = vessel diameter; π = the value of 3.14 Ls = cylinder height	(2)
		The volume of Liquid in The Cylinder (V_{ls})	V_{ls} = V liq + V top lid $V_{ls} = V\ liq\ \frac{\pi di^3}{24 \tan 1/2\alpha}$ V_{ls} = the volume of liquid in the cylinder Di = vessel diameter; π = the value of 3.14	(3)
		High Liquid in The Cylinder (L_{ls})	$L_{ls} = \frac{V\ ls}{\left(\frac{\pi}{4}\right) x\ di^2}$ L_{ls} = high liquid in the cylinder V_{ls} = the volume of liquid in the cylinder π = the value of 3.14 Di = vessel diameter	(4)
		Design Pressure (Pi)	Pi = P atm + P hydrostatic P hydrostatic = $\frac{P(HL-1)}{144}$ + C	(5)
		Cylinder Thickness (Ts)	$Ts = \frac{Pi\ .di}{2(f.E-0{,}6Pi)} + C$ Ts = cylinder thickness Pi = design pressure Di = vessel diameter	(6)
		Cylinder Height (Ls)	Vtotal = V top lid + Vcylinder + V top cov V total = $\frac{\pi di^3}{24 \tan 1/2\alpha} + \frac{\pi di^2}{4}$ x Ls + 0,0847 di^3 Di = vessel diameter; π = the value of 3.14 Ls = cylinder height	(7)
		Top Cover Thickness (tha)	tha = $\frac{0{,}885 \times Pi.di}{2(f.E-0{,}6Pi)}$ + C tha = top cover thickness; Pi = design pressure; Di = vessel diameter	(8)
		Top Cover Height (ha)	ha = 0,169 Di ha = top cover height; Di = vessel diameter	(9)

		Bottom Cover Thickness (thb)	thb = $\frac{\mathrm{Pi\,.\,di}}{2(\mathrm{f.E}-0{,}6\mathrm{Pi})\cos 1/2\alpha}$ + C Pi = design pressure; Di = vessel diameter	(10)
		Bottom Cover Height (hb)	hb = $\frac{1/2\,d}{\tan 1/2\alpha}$	(11)
2.	Reactor Stirrer	Impeller Diameter (Da)	Da = Dt × 0.5 Da = impeller diameter Dt = cylinder inside diameter	(12)
		Impeller Height from Tank Bottom (C)	C = $\frac{1}{3} \times Di$ C = cylinder inside diameter Di = vessel diameter	(13)
		Impeller Length (L)	L = $\frac{1}{4} \times Da$ L = impeller length Da = impeller diameter	(14)
		Impeller Width (W)	W = 0.20 × Da W = impeller width; Da = impeller diameter	(15)
		Number of Stirrer (n)	n = $\frac{\mathrm{H\ liquid}}{2 \mathrm{\ x\ Da}^5}$ n = the number of stirrer; Da = impeller diameter	(16)
		Reynold Number (N_{Re})	$N_{Re} = \frac{\mathrm{L}^2 \mathrm{\ x\ n\ x\ } \rho}{\mu}$ N_{Re} = the Reynold number L = impeller length n = stirrer rotation, set = 100 rpm = 1,67 rps ρ = density (lb/ft^3)	(17)
		Stirring Power (P)	P = $\frac{\varphi \times \rho \times n^3 \mathrm{\ x\ } Di^5}{gc}$ P required = (0,1 + 0,15)P + P P = stirring power; ρ = density (lb/ft^3) Di = impeller diameter; gc = 32,2 lb.ft/s^2.lbf	(18)
		Stirrer Shaft Diameter (D)	D = $\frac{\#\ x\ T}{\pi\ x\ S}$ D = stirrer shaft diameter T = torsion number (lb.in = $\frac{63025\,H}{N}$) π = the value of 3.14 S = maximum allowable design shearing stress	(19)
		Shaft Length (L)	$L = h + l - Zi$ h = cylinder height + top cover height l = impeller distance from the tank bottom Zi = length of the shaft above tank vessel	(20)

RESULTS AND DISCUSSION

Main Reaction

In this research, the production of zinc oxide (ZnO) was carried out using a Continuous Stirred Tank Reactor (CSTR) type reactor. The precursor compounds synthesizing ZnO nanoparticles are NH_4HCO_3 and $ZnSO_4·7H_2O$. The reactions that occur in this production process are shown in equations number (1) and (2).

(1). $5ZnSO_4\ (aq) + 10NH_4HCO_3\ (aq) \rightarrow Zn_5(CO_3)_2(OH)_6\ (s) + 5(NH_4)_2SO_4\ (aq) + 8CO_2\ (g) + 2H_2O\ (l)$

(2). $Zn_5(CO_3)_2(OH)_6\ (s) \rightarrow 5ZnO\ (s) + 2CO_2\ (g) + 3H_2O\ (g)$

The above reaction shows that when NH_4HCO_3 is reacted with $ZnSO_4·7H_2O$ it will produce solid white ZnO as the main product and white solid $(NH_4)_2SO_4$ as a side product.

Reactor Type

A reactor is a process tool where a reaction occurs, both in small sizes, such as test tubes and large sizes, such as industrial-scale reactors. In this study, the reactor was used as a place for the reaction between NH_4HCO_3 and $ZnSO_4·7H_2O$ to produce white ZnO nanoparticles as the main product. The reactor used in this study was the CSTR type in the form of an upright cylinder with a standard dished top lid and a conical bottom lid with a peak angle of 120° and using SA 240 Grade M Type 316 stainless steel. In the CSTR-type reactor, parameters such as temperature, concentration, and the reaction rate between the reacting substances will be the same at all reactor positions. In controlling a reactor system, several controls are needed to be placed on the CSTR reactor, one of which is a pressure controller. High and continuous pressure in the system can cause an explosion, so the pressure in a closed system needs to be maintained by controlling the pressure in the reactor to keep it safe.

In addition to controlling pressure, stirrers are crucial in manufacturing CSTR-type reactors. The success of a treatment process often depends on the effective mixing of the liquids in the process. Agitation is the reduction of movement in a certain way in a material in a vessel, where the movement usually has some circulation. The purpose of stirring is to create a suspension of solid particles, dissolve liquids that cannot be mixed with other liquids, form fine-grained emulsions or suspensions, and accelerate heat transfer between the liquid and the mantle. Mixer is expected to produce the best mix with the lowest possible power. In CSTR, reactants and products flow continuously. Raw materials are continuously added, and reaction products are continuously removed. To achieve uniform composition and temperature, CSTR requires mechanical or hydraulic stirring. The ideal reactor for CSTR can be described with perfect stirring conditions to produce a well-mixed reaction mixture. Perfect mixing is required so that the composition and temperature are uniform at all points, assuming the density does not change (negligible) because the volume does not change to achieve high homogeneity.

Reactor Parameter Calculation Results

The results of mass balance calculations can be used to select the appropriate tool and size and provide volume for the process [26]. Table 2 shows the results of mass balance calculations in ZnO production.

Table 2. Recapitulation of the mass balance of ZnO production

Component	Mr (g/mol)	Reactants			Product		
		Massa	Mol	Fr.Mol	Mol	Fr.Mol	Massa
$Zn_5(CO_3)_2(OH)_6$	548.96	4742.840	8.640	0.044	0.432	0.001	237.142
5ZnO	406.90	5435.420	13.358	0.069	76.810	0.113	31253.666
$2CO_2$	88.02	5850.968	66.474	0.342	192.774	0.285	16967.807
$3H_2O$	54.05	5712.452	105.696	0.544	406.931	0.601	21992.941
Total		21741.680	194.168	1.000	676.947		70451.555

The data shows that the mass of $Zn_5(CO_3)_2(OH)_6$ that enters is 4742.840 kg/h. In contrast, the ZnO, CO_2, and H_2O masses that came out were 5435.420, 5850.968, and 5712.452 kg/h. The total product mass obtained from the reactor is 70451.555 kg/h.

Next, the calculation of the dimensions of the reactor is carried out. The type of reactor used is an upright cylinder with a standard dish top lid and a conical bottom lid with a peak angle of 120°. The condition of the reactor for the manufacture of ZnO nanoparticles was at a temperature of 100°C, a pressure of 1 atm with an operating time of 1 hour, had an allowable stress (f) of 18750, double-welded butt joint E of 0.8, and a corrosion factor of 0.0625. Table 3 shows the results of calculating the general dimensions of the reactor.

Table 3. Reactor dimension specifications based on calculation results

No.	Parameter	Result
1.	Type of reactor	Upright cylinder with standard dished top cap and conical bottom cap with a peak angle of 120°
2.	Volume Reaktor (V_{total})	8224.359 liter
3.	Vessel Diameter (Di)	73.298 in
4.	The volume of Liquid in The Cylinder (V_{ls})	6114.993 liter
5.	High Liquid in The Cylinder (L_{ls})	2505.423 in
6.	Design Pressure (Pi)	4.304 psig
7.	Cylinder Thickness (Ts)	73.444 in
8.	Cylinder Height (Ls)	166.090 in
9.	Top Cover Thickness (tha)	0.072 in
10.	Top Cover Height (ha)	12.387 in
11.	Bottom Cover Thickness (thb)	0.083 in
12.	Bottom Cover Height (hb)	21.185 in
13.	Reactor Height	37.552 in

The results of calculating the reactor dimensions obtained a reactor volume of 8224.359 liters, with a vessel diameter of 73.298 in, a cylinder height of 166.090 in, and a cylinder thickness of 73.444 in. After getting the diameter of the vessel, the next calculation is to calculate the height of the top cover and the bottom cover. Such calculations will yield an overall high result. The result of the top cover calculation is 12.387 in with a thickness of 0.072 in, while the result of the bottom cover calculation is 21.185 in with a thickness of 0.083 in. The overall height of the reactor is 37.552 in.

The dimensions of each component, including the reactor's stirrer calculation, also need to be considered. Stirrer also called an agitator, generally consists of a series of motors as a drive pad and an impeller or blade adapted to the organic materials used. The existence of stirring in the process of forming ZnO nanoparticles will form a flow pattern in the reactor. The type of stirrer to be used is an axial turbine with 4 blades at an angle of 45° with impeller construction made of High Alloy Steel SA 240 Grade M type 314, and the material used for the construction of the stirrer shaft is Hot Rolled Steel SAE 1040. After selecting the stirrer plan, then calculate the dimensions. The stirrer to be used and the calculation results are presented in Table 4.

Table 4. Specification of stirring dimensions based on calculation results

No.	Parameter
1.	Impeller Diameter (Da)
2.	Impeller Height from Tank Bottom (C)
3.	Impeller Length (L)
4.	Impeller Width (W)
5.	Number of Stirrer (n)
6.	Reynold Number (N_{Re})
7.	Stirring Power (P)
8.	Stirrer Shaft Diameter (D)
9.	Shaft Length (L)

The calculation obtained the number of stirrers 1 unit with an impeller diameter of 36.722 in, impeller height from the bottom of the tank of 24.433 in, impeller width of 7.344 in, and impeller length of 9.180 in. It is known that the plate used in the stirrer is an axial turbine type with 4 blades angle of 45°. The turbine stirrer type is a type of stirrer that has many blades and is shorter in size. This type of stirrer is used at high speeds for liquids over a very wide range of viscosities. The turbine agitator diameter is 30 - 50% of the tank diameter. In turbines with 45° inclined blades, several axial flows will be generated to form a combination of axial and radial flows. This type is useful in the suspension of solids because the flow is directed downward and will sweep the solids upward.

The amount of input power obtained is 6.849 Hp. Mixing power utilizes the occurrence of chemical reactions from raw materials to the desired product. The power of this stirrer will affect the magnitude of the resulting velocity gradient. Stirring power is generated by the mixing system, for example, the stirrer and its rotational speed, water flow, airflow, and so on. In the calculation of the stirrer shaft with a shaft power of 6.849 Hp with a stirrer rotation of 100 rpm, the diameter of the stirrer shaft is 1.070 in, and the length of the shaft is 163.224 in. The shaft is a transmission of power or torque, usually round in cross-section. Based on the literature, the material used to manufacture the shaft must be selected, which is corrosion-resistant [26]. Therefore the shaft material used in this design is Hot Rolled Steel SAE 1040, which is corrosion-resistant.

In addition, the Reynolds number is 99796.530. The Reynolds number is the ratio between inertia and viscosity. Processes with mechanical agitators occur under laminar or turbulent flow conditions, depending on the Reynolds number of the impeller. The type of flow that occurs here is turbulent flow because of the value of Re > 2100. Turbulent flow is a fluid flow in which the particles move randomly and are unstable, which causes the flow lines between the fluid particles to intersect. The opportunity for materials to interact or collide is greater than in laminar conditions, namely fluid conditions that move statically and regularly without crossing each other. The agitator with this type of turbulent flow provides the best possible mixing.

CONCLUSION

Based on the calculation of the reactor design, the obtained reactor volume is 8224.359 liters, with vessel diameter of 73.298 in, cylinder height of 166.090 in, and cylinder thickness of 73.444 in. The top cover of the reactor measures 12.387 inches with a thickness of 0.072 inches while the bottom cover measures 21.185 inches with a thickness of 0.083 inches. So the overall height of the reactor is 37.552 in. The reactor is equipped with one stirrer with an impeller diameter of 36.722 in, impeller height from the bottom of the tank is 24.433 in, impeller width is 7.344 in, and impeller length is 9.180 in. Turbulent stirring flow conditions with a standard motor power for the stirrer is equal to 6.849 HP. This design is expected to be a reference for building more economical, efficient, and high-demand reactors.

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