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Design and Fabrication of a Multi-tubular Fixed Bed Reactor for Acetone Production as A Pilot Plant Model for Chemical Engineering Training in Developing Nations

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Abstract

In this work, the design and fabrication of a pilot-scale multi-tubular fixed bed reactor have been carried out with the aid of computational software (MATLAB R2010b), aiming to process 6.5 L/h of isopropanol to acetone using non-adiabatic thermal conditions. The reactor fabricated would serve as a demonstration rig to enhance research practice, teaching, and learning processes in Advanced Reactor Analysis. The acetone production studied involves four stages, which are simultaneous vaporization and dehydrogenation, condensation, and storage. In the simultaneous vaporization and dehydrogenation, isopropanol is heated to 473 K indirectly to change the phase of raw material from liquid to vapor. In comparison, isopropanol is dehydrogenated to be acetone and hydrogen with 50 % conversion using Cu/SiO₂ composite as a catalyst in the multi-tubular fixed bed reactor at 473 K and 1 bar. Condensation occurs when an ice bath cools down the vapor product to liquid. Hence, the condensed product is tested for acetone, and the overall cost analysis for the whole process is calculated. The design can be a model for other chemical engineering schools to fabricate a locally made reactor for the training and re-training of chemical engineers, especially in developing nations.

Keywords: Reactor Design, Chemical Reaction Engineering Education, Multi-tubular Fixed-Bed Reactor, Fabrication.

1. Introduction:

Acetone (propanone) is an organic compound with the formula CH₃ [1]. It is a colorless, volatile, flammable liquid and is the simplest of the ketones. Acetone is miscible with water and has been reported to be one of the most essential solvents typically used for cleaning purposes in laboratories.

About 6.7 million tonnes were produced worldwide in 2010, mainly for use as a solvent and production of methyl methacrylate and bisphenol A [2]. It is a common building block in organic chemistry for the design of new materials or chemicals. Everyday household uses of acetone are the active ingredients in nail polish remover and paint thinner.

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Acetone has been an excellent solvent for various greases, oils, gums, waxes, dyestuffs, resins, fats, and cellulosic. It is used as a carrier for acetylene, in the manufacture of a variety of coatings and plastics, and as a raw material for the chemical synthesis of a wide range of products such as ketene, methyl methacrylate, bisphenol A,

diacetone alcohol, methyl isobutyl ketone, hexylene glycol (2-methyl-2,4-pentanediol), and isophorone [3]. The comparison of different process routes for the production of acetone obtained from the literature is summarised in Table 1. From which the chosen process route was justified.

Table 1. Comparison between processes and selection of process

Cumene process [4]-[6]	Propylene oxidation process [7]	Isopropyl alcohol dehydrogenation process [8], [9]
The primary product is phenol Acetone is a by-product The purity of acetone is low. ---	Acetone is the primary product Purity is high The propylene used should be 99% pure.	Acetone is the primary product Purity is high An aqueous solution of the isopropyl alcohol can be used.
Conversion to acetone is low. Worldwide production method Unconverted benzene present along with acetone is dangerous to some processes.	Conversion to acetone is low Not used worldwide No dangerous compound present along with acetone	Conversion to acetone is high Worldwide production method No dangerous compound present along with acetone
Many separation processes are required to purify acetone, which increases the production cost confirmation.	Less separation process is required, and production cost is low	Less separation process is required, and production cost is low

The major disadvantage of producing acetone from the cumene process [10], [11] is that some reactant benzene is present along with the desired product, which is toxic. In contrast, the challenge of producing acetone from the propylene oxidation process [12] is that the propylene required for the operation should be 99 % pure. However, the process our study deployed for making acetone was a process that dehydrogenates isopropyl alcohol in the presence of a solid catalyst [13][16], a lesser disadvantage than other methods. Moreover, the process for this design chosen was considered to be satisfactory due to its demand for more secondary pressure of 1 bar and a moderate temperature of 200 °C, which is obtainable at the laboratory/pilot scale level for teaching and demonstration in Advanced Reactor Analysis or related courses in Ahmadu Bello University (ABU), Zaria.

A literature survey [4][9], [17], [18] indicated that much work had been carried out on acetone production. Some of these works include Tohaneanu et al. [3] that simulated the production

of acetone from isopropanol using Cu/SiO₂ catalyst; Said et al. [19] employed the use of Cu-NiO, which indicated that the introduction of NiO decreases the activation energy of conduction. Unlike other works, Balouch et al. [20] employed palladium to investigate the hydrogenation of acetone into isopropanol. Ramachandriya et al. [21] employed *Clostridium ragsdalei* to reduce acetone to isopropanol. Some studies employed the use of biocatalysts. One of these studies includes Jojima et al. [22], who employed *Escherichia coli*. to produce isopropanol from the use of glucose. However, the motivation behind our studies was the absence of a multi-tubular fixed bed reactor set-up rig that could be used for the effective teaching and research of Advanced Reactor Analysis in the Department of Chemical Engineering, Ahmadu Bello University Zaria in Nigeria.

The challenge, therefore, geared the studies' aim toward designing and fabricating a multi-tubular fixed-bed reactor for producing acetone from isopropanol over a copper-silica catalyst, which

could be used for research and teaching demonstration in ABU Zaria. This aim was achieved by developing a process flow diagram, choosing a design basis, and collecting relevant data necessary for the analysis. The volume and weight of catalyst in the tubes, the tubes, shell, pitch, and baffles size presented in the reactor, and the heat load or duty required to drive the operation of the pilot plant were determined. In addition, the cost analysis of the pilot plant was investigated. The data needed in this design were sourced from literature (papers, textbooks, and internet resources). Assumptions were made where specific information was not available. The design plan was strictly limited to converting 6.25 L isopropanol to acetone using a catalyst (CuSiO_2) per hour for a square pitch and non-adiabatic operation. This study work adopts the approach of both a multi-tubular reactor and shell and tube heat exchanger model. The computational analysis involved was aided by using a MATLAB computational tool.

2. Materials and Methods:

2.1 Materials:

The materials used include isopropanol, catalyst (copper nitrate over silicon oxide), heavy oil, ice water, and glass fiber.

2.2 Reaction kinetics for the process:

According to the literature [23], the reaction can be controlled by selective catalysts and proper operating conditions. The primary reaction for producing acetone is:



And the kinetics for this reaction is given below:

$$-R_{\text{IPA}} = kC_{\text{IPA}} \text{ in } \text{m}^3 \text{ gas}/\text{m}^3 \text{ cat} \quad (2)$$

where, k is the reaction rate constant obtained from literature as $1.76 \times 10^5 \exp(-60,000/(T)/8.314)$; C_{IPA} is isopropanol concentration expressed in mol/m^3 gas. Although several side reactions are possible, none of them occurs considerably. The activation energy in the kinetic expression above is in units of kJ/kmol [23].

2.3 Catalyst Preparation & Characterization

2.3.1 Catalyst preparation:

The preparation of the catalyst was split into three batches. Each batch was prepared the same way. A

solution of silica powder (Fischer), 48g, was prepared using deionized water and heated up to 70°C . A solution of 0.748 molarity Cu made from $\text{Cu}(\text{NO}_3)_2$ (JDH) was added dropwise to the silica solution. The mixture was heated with constant stirring to dryness at 70°C , which took roughly 8 hours. The resulting powder was dried overnight at 70°C . The powder was then calcined at 600°C for 3 hours before use as a catalyst.

2.3.2 Catalyst characterization:

Fourier transforms infrared spectroscopy was conducted using the Shimadzu 8400S FTIR analyzer (insert proper model). The analysis observed differences between the pristine and copper-impregnated silica support. Crystallographic properties of the synthesized catalyst were analyzed using a Rigaku mini flex 300 X-Ray diffractometer with two thetas ranging from 5 - 80° .

2.4 Process Description And Route:

The process route is shown in Figure 1. It shows the process flow of the materials from the raw material tank (U-RT) to the product tank (U-PT) to produce acetone from isopropanol. In this simplified process, an aqueous solution of isopropanol from the Raw Material Tank (U-RT) was transported to the multi-tubular fixed bed reactor (U-MTFBR) with a pump to maintain the pressure. At the same time, a control valve (CV) was used to regulate the flow rate of the isopropanol in-stream S-2c at a temperature of 25 - 35°C and a pressure of 1 atm.

Each tube in the reactor (U-MTFBR) contains a fixed bed of catalysts (copper silica). Engine oil was chosen as a heat transfer medium due to its relatively high heat capacity and good heat transfer properties. The oil is heated to a temperature of 250°C in the reactor's shell (U-MTFBR) from the immersed heater. This, in turn, heats the tubes containing the catalyst to about 200 - 220°C . The isopropanol vapor is dehydrogenated as it passes through the reactor (U-MTFBR) and over the copper silica catalyst at 200°C and 1atm to form Acetone; stream S-3 is a gaseous phase product at that temperature.

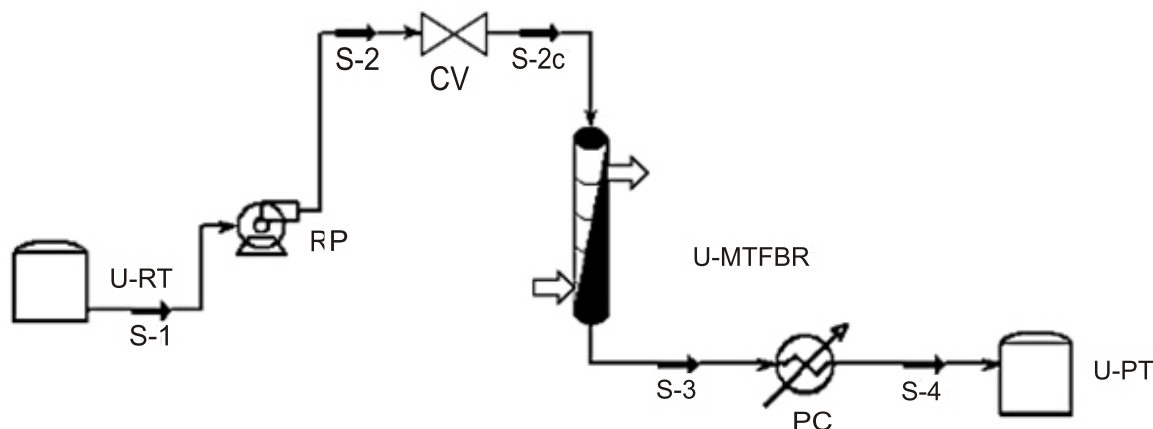


Figure 1. Process Flow Diagram for the Production of Acetone from Isopropanol (Key: U-RT = Raw material tank, U-PT = Product tank, U-MTFBR = multi-tubular fixed bed reactor, RP = Raw material pump, CV = Control valve, PC = Product condenser, S-1=Isopropanol, S-2=Pumped isopropanol, S-2c=Regulated isopropanol, S-3= Fresh acetone, S-4=Cooled acetone)

It then passes into a condenser (PC), which cools down the gaseous product in stream S-3 into liquid product stream S-4, stored in the product tank (U-PT). The product collected is not pure acetone due to unreacted isopropanol (a conversion of roughly 50% is expected). So, a suitable chemical analysis is used to identify the presence of acetone in the product mixture (Fehling's test, the Iodoform test, and the Sodium bisulfite test). Figure 1 displays the graphical illustration of the process flow diagram.

2.5 Reactor Design:

The computation analysis of the multi-tubular fixed-bed reactor design was carried out using MATLAB computational software. The reactor was designed to convert 6.5 L/h (2.5 L/0.4h) of isopropanol into acetone using a solid catalyst of copper nitrate over silicon oxide, non-adiabatic operation, catalyst in a tube, 6 number of tubes, two tube passes, one shell pass, external tube diameter 17.1 mm, tube thickness of 2.2 mm and square pitch at 200 °C and 1 bar.

2.5.1 Basic data used in the design:

A MATLAB algorithm (please, see the details on codes and parameters in Supplementary Material) was written to compute various relevant parameters for the sizing of the setup equipment necessary for designing and fabricating the multi-tubular fixed bed reactor setup. The reactor volume (V_p) was taken as 2 L using six tubes (i.e., $N_t = 6$), the set desired reaction conversion (X) was 50 %, the

reaction time (t_p) was 1 hour, the reactor flowrate (V_r) was computed as " V_p/T_p ", and the corresponding mass flowrate (M_{ao}) were calculated as the product of the volumetric flowrate (V_r) and the isopropanol fluid density (den_f). The tube mass flow rate (M_{aot}) was calculated as " M_{ao}/N_t ". The molar flow rates were also computed. These are programmed in the preliminary section of the MATLAB algorithm (presented in the supplementary information) for the computations.

2.5.2 Estimation of volume and weight of catalyst per tube:

The weight, W_t , and volume, V_t , of the catalyst per tube were estimated using the rate expression from Missen *et al.* [24] in the form which was solved via the use of the Simpson one-third rule:

$$V_t = F_{aot} \int_a^b \frac{1}{r_A} dX_a \quad (3)$$

$$r_A = kC_a = kC_{a0}(1 - X_a) \quad (4)$$

$$V_t = \frac{F_{aot}}{kC_{a0}} \int_a^b y dX_a, \text{ where } y = 1 - X_a \quad (5)$$

The solution of the above numerical integration expression via the use of Simpson's one-third rule and integral boundary of 0 % (i.e. 'a') to 50 % (i.e. 'b') in space interval of 100 steps with the aid of MATLAB-based solvers and looping commands that was deployed in the algorithm.

$$V = V_t * N_t \quad (6)$$

$$B_{den} = C_{den} * (1 - B_v) \quad (7)$$

$$W_t = V_t * B_{den} \quad (8)$$

$$W = W_t * N_t \quad (9)$$

Where the weight of catalyst in each tube (W_t) and the total required catalyst weight (W) were obtained using relevant data like catalyst density (C_{den}) of 2.34 g/cm³, particles diameter (d_p) of 15 mm, bed voidage (B_v) of 0.64 and catalyst voidage (cv) of 0.4.

2.5.3 Sizing of reactor tubes:

Based on the volume and weight of the catalyst, the reactor tubes present in the reactor were sized [24], [25] in relation to the results collected for the volume (V) and weight of the catalyst (W) required in a tube including the chosen tube diameter (D_t) as 10 mm and thickness (t_n) as 0.0022 mm (with reference to what is obtainable in the market). The analysis involves the determination of tube length (L_t) and pressure drop (dP) in tubes, including the estimation of the tube pitch (P_t) and bundle diameter (Db) that would be required for the fixing of the tube in the reactor's interior. The MATLAB algorithm aided the analysis (as presented in the Supplementary Information). The tube's internal diameter was computed as " $D_t - 2 * t_n$ " and the tube length (L_t) was computed as:

$$L_t = (tr + 1) * 4 * \frac{W_t}{B_{den} * \pi * D_t^2} \quad (10)$$

Pressure drop (dP) in tubes was computed using:

$$dP = L_t * \left(150 * \frac{\rho}{B_v * d_p} + 17.5 * G \right) * \left(\frac{(1 - B_v)^2}{B_v^2} \right) * \left(\frac{G}{F_{den} * d_p} \right) \quad (11)$$

Where the cross-sectional area in m² is $A_{it} = \pi * (D_{ti})^2 / 4$, mass velocity in kg/sec/m² is $G = M_{aot} / A_{it}$. The pressure drop in tubes (dP) obtained was assessed to be less than 3 bar for a good design [24], [25]. The bundle diameter is computed using the number of tubes (N_t), square pitch, two tube passes, and one shell pass.

$$P_t = 2.50 * D_{to} \quad (12)$$

$$T_{sp} = (P_t - D_{to}) * 1000 \quad (13)$$

$$D_b = D_{to} * \left(\frac{N_t}{K_1} \right)^{\frac{1}{n_1}} \quad (14)$$

The calculation for the Bundle diameter (Db) was done using $K_1 = 0.156$, $n_1 = 2.291$ for the chart with two tube passes and square pitch. The results obtained for the computation were collected for the relevant parameters of interest, which include the tube pitch (P_t), tube spacing (T_{sp}), bundle diameter

(Db), and tube length (L_t).

2.5.4 Estimation of the heating duty

With reference to Sinnott *et al.* [26] the duty of the reactor's heating medium that would be required to provide the condition for the reaction within the multi-tubular reactor was estimated with the use of the MATLAB-based algorithm (presented in the supplementary information) shown below, which computes the heat load or duty (Q) including the overall transfer coefficient (U) and tube heat transfer coefficient, ht .

$$Q = n * \Delta H_{to} - H_{ti} \quad (15)$$

Where H_{to} is the enthalpy at the tube outlet while the H_{ti} is the one obtained at the tube inlet, $n = F_{a0}$ is the molar flow rate. The log-mean temperature (dT_{lm}):

$$dT_{lm} = \frac{(T_{bo} - T_{bi}) - (T_{si} - T_{so})}{\ln \left(\frac{T_{bo} - T_{bi}}{T_{si} - T_{so}} \right)} \quad (16)$$

$$R = \frac{T_{si} - T_{so}}{T_{bo} - T_{bi}} \quad (17)$$

$$S = \frac{T_{bo} - T_{bi}}{T_{si} - T_{bi}} \quad (18)$$

T_{so} is the shell temperature outlet, T_{si} is the shell temperature inlet, T_{bi} is the tube temperature inlet, and T_{bo} is the outlet. Taking correction factor (F_t) from the chart using R and S to obtain corrected mean temperature (dT_m) and overall transfer coefficient (U):

$$U = \frac{Q}{dT_m * F_t * A} \quad (19)$$

Where $A = N_t * \pi * D_{to} * L_t$ and $dT_m = dT_{lm} * F_t$. The results were collected for the heat load, overall heat transfer, and tube transfer coefficient.

2.5.5 Sizing of shell and baffles:

Other components of the multi-tubular fixed-bed reactor sized were the shell and baffles using the relevant data provided in the earlier section of this report in the computation of shell volume (V_s), shell diameter (D_s), shell length (L_{sh}), baffle spacing (B_s), the number of baffles required in the reactor (N_b), crossflow area (A_s), shell equivalent diameter for the chosen square pitch (in line with the basis for the design) (D_e), shell heat transfer coefficient (h_s), and shell pressure drop (dP_s). These computations were carried out using some relevant models and/or with the help of relations adopted from Sinnott *et al.* [27], as well as with the aid of the MATLAB algorithm (presented in the supplementary information) for enhancement of calculation. The

results obtained from the design specification required for the fabrication of the reactor were collected.

3. Results and Discussion:

3.1 FTIR and XRD analysis of synthesized CuSiO_2 catalyst:

FTIR analysis of the synthesized catalyst is shown in Figure 2. Spectra of the pristine silica support were juxtaposed with the copper-impregnated catalyst. The peak emanating at 1095 cm^{-1} was ascribed to the asymmetric stretching vibrations of the Si-O-Si band in amorphous SiO_2 , which was similar to the findings made from the studies of Peña-Alonso et al. [28] and Ye et al. [29] who reported 1103 cm^{-1} and 1070 cm^{-1} respectively as an asymmetric stretching of Si-O-C. Further analysis of the spectra indicates that the peak at 811 cm^{-1} represents the symmetric stretching of the Si-O-Si bond. The early peak at 460 cm^{-1} is due to bending vibration between silica and oxygen links. In comparison, the prominent peak at 3400 cm^{-1} is attributed to Si-OH asymmetric stretching vibrations alongside some possible contribution from adsorbed OH of water which typically presents peaks at 3600 cm^{-1} . [29]. The presence of the peak at 1635 cm^{-1} confirms the contribution of adsorbed OH from water as a result of moisture trapped in the silica matrix. The weak shoulder at 956 cm^{-1} is due to the formation of copper phyllosilicates [13] and the Cu-O-Si bending vibration [28]. The spectra of SiO_2 and CuSiO_2 also exhibit a difference in the shape of the spectra at 568 cm^{-1} [30]. This deviation in the pattern is due to the formation of CuO after calcination [31].

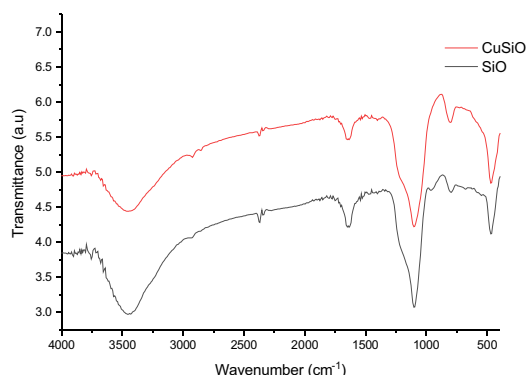


Figure 2. FTIR of the catalyst Copper-supported by Silica

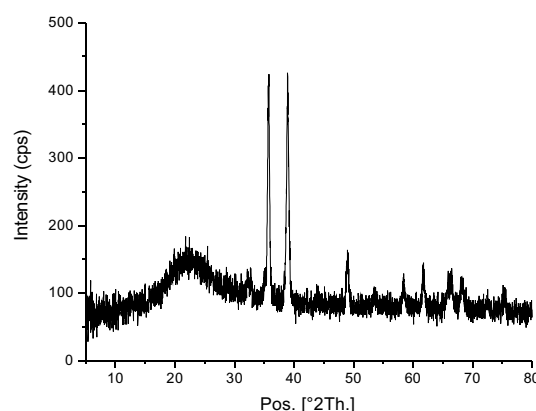


Figure 3. XRD Diffractograms of Synthesized CuSiO_2 Catalyst

Figure 3 shows the XRD pattern of the synthesized CuSiO_2 catalyst. The amorphous peak associated with SiO_2 was observed at a 2θ value of approximately 22.6° . Cu was present in the form of CuO in agreement with the FTIR spectra shown in Figure 2. The seven most prominent peaks were used to determine the crystallite size using the Scherrer equation. The peaks had two theta values of 35.7° , 38.8° , 49.0° , 58.4° , 61.7° , 66.3° , and 68.3° . CuO had an average crystallite size of 28.47 nm . Peaks at 35.78° and 38.9° are a result of the CuO tenorite phase, which was found to agree with the report of Reinosa et al. [32] which also obtained peaks at 35° and 39° . Also, a minuscule peak at 32.18° is ascribed to the presence of copper phyllosilicate, agreeing with the peak at 956 cm^{-1} observed in the FTIR. The low visibility of this peak suggests a very low concentration of phyllosilicates is present in the structure.

3.2 Results of Reactor Design:

The results obtained from the design of the reaction are presented in Table 2, displaying the number of tubes, tube dimensions, shell dimensions, tube arrangement in the shell, and amount of catalyst to be loaded in each tube of the reactor, including the pressure in both the shell and the tubes.

Table 2: Results obtained from the design of the reactor

Parameter	Notation	Unit	Values
Baffle spacing	Bs_cm	cm	6.2112
Bundle diameter	Db	m	0.0841
Conversion	X	%	50
Heat transfer area	A	m ²	0.0809
Heating fluid duty/load	Q	kJ/s	1.3811
Length of the shell	Lsh_cm	cm	35.9021
Length of tube	Lt_cm	cm	25.1063
The mass flowrate of isopropanol	Fao	kg/s	2.2040e-005
Number of baffles	Nb		3.0421
Number of tubes	N_tube	tube	6
Outer diameter of tube	Dto	mm	0.0171
Overall heat transfer coefficient	U	kW/m ² .°C	0.1084
Overall shell mass flow rate	Ms_overall	kg/s	6.8841
R correction factor (from the chart)	R	-	0.9091
Reaction temperature	T_K	K	473
Reaction time length	tp	S	1440
S correction factor (from the chart)	S	-	0.7674
Shell diameter	Ds_cm	cm	9.4110
Shell equivalent dia. for square pitch	De	cm	11.8683
Shell heat transfer coefficient	hs	W/m ² .°C	1.7597
Shell pressure drop	dPs_bar	bar	2.6372e-008
Shell volume	Vsh	m ³	0.0025
The total volume of catalyst required	V	m ³	1.7348e-004
Total weight of catalyst required	W_kg	Kg	0.1461
Tube heat transfer coefficient	ht	W/m ² .°C	3.5764
Tube internal diameter	Dti	mm	0.0127
Tube mass flow rate of isopropanol	Faot	kg/s	3.6733e-006
Tube pitch	Pt_mm	mm	42.7500
Tube pressure drop	dPt_bar	bar	0.0018
Tube spacing	Tsp_mm	mm	25.6500
Tube volume	Vt	m ³	2.8913e-005
Volume of isopropanol to be processed	Vp	m ³	0.0025
Volumetric flow rate	Vr	(m ³)/s	1.7361e-006
Weight of catalyst per tube	Wt	Kg	0.0244

The obtained equipment design outcome was found to be within the acceptable standard range where the reported pressure for the shell and tube flows were both less than 3 bar in line with existing reports in the literature [24], [33]. The heat load (or the heating fluid duty or load) was estimated to be 1.3811 kJ/s.

3.2.1 Estimation of volume and weight of catalyst per tube:

The analysis computation results (as shown in Table 2) obtained for the volume, Vt, and weight,

Wt, of the catalyst required to be present in each tube fixed in the reactor were recorded as 2.8913e-005 m³ (28.913 mL) and 0.0244 kg (24.4 g). The results indicated that the reactor would require a total catalyst weight of 0.1461 kg, which was found to be reasonably well for the training and research purpose as its material demand for the teaching or consumable research materials would be minimal. And it would be affordable for the institutes in the developing world to operate and manage its use.

3.2.2 Sizing of reactor tubes and shell:

The results obtained for the computation were collected for the relevant parameters of interest, which include the tube pitch, bundle diameter, and tube length as 42.75 mm, 0.084 m (8.4 cm), and 25.11 cm, as shown in Table 2. The results obtained for the shell volume, length, and diameter required for the fabrication of the reactor, as shown in Table 2, indicated 0.0025 m³ (2.5 L), 35.9021 cm, and 9.41 cm, respectively.

3.2.3 Sizing of heat transfer coefficient and baffles:

The results obtained for the overall heat transfer

coefficient, shell heat transfer coefficient, tube heat transfer coefficient, the baffle spacing, and the number of baffles required for the fabrication of the reactor, as shown in Table 2, indicated 1.7597 W/m².°C, 3.5764 W/m².°C, 0.1084 kW/m².°C, 6.21 cm spacing, and three baffles are required, respectively.

3.3 Equipment Specifications:

From the design carried out, the equipment specifications and fabrications are presented in Table 3. The specifications were used in fabricating the pilot plant setup using the engineering drawings provided in Figures 4 and 5.

Table 3: Multi-tubular Fixed Bed Reactor Fabrication Specifications

Description	Symbol	Units	Values
Baffles Cutting	-	%	25
Baffles Spacing	-	Mm	621.1
Number of Baffles	Nb	Baffles	3
Number of tubes	Nt	-	6
Pitch type	-	-	Square
Shell Internal diameter	Ds	cm	9.41
Shell Length	Ls	mm	359
Shell pass		Passes	1
Shell Pressure drop	dPs	Bar	2.6372e-008
Tube external diameter	Dto	mm	17.1
Tube head type	-	-	Fixed
Tube Length	Lt	mm	251
Tube pass	-	Passes	2
Tube Pressure drop	dPt	Bar	0.0018
Tube thickness	Tn	mm	2.2

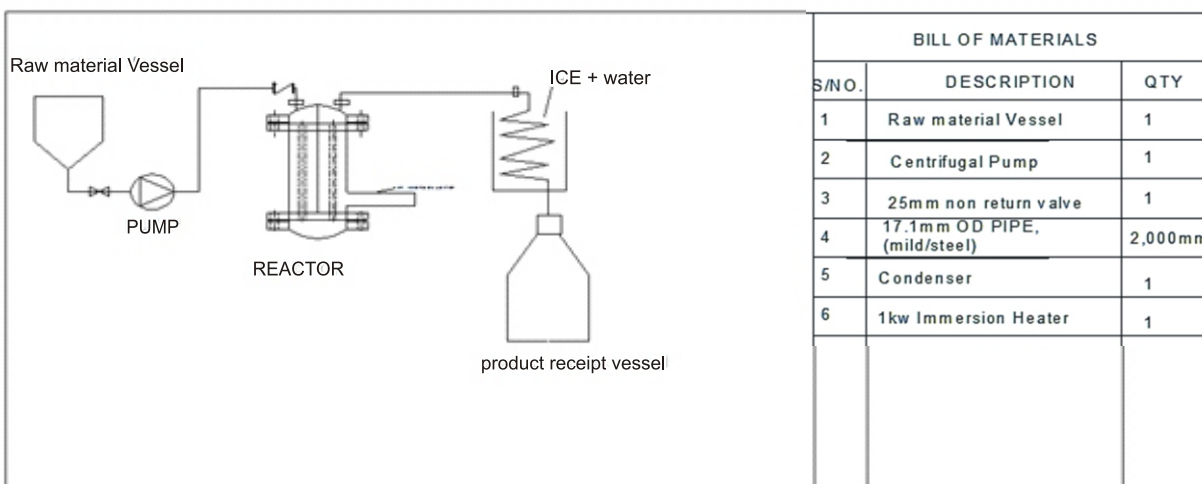


Figure 4: Two-Dimensional Drawing of the Set-up Arrangement

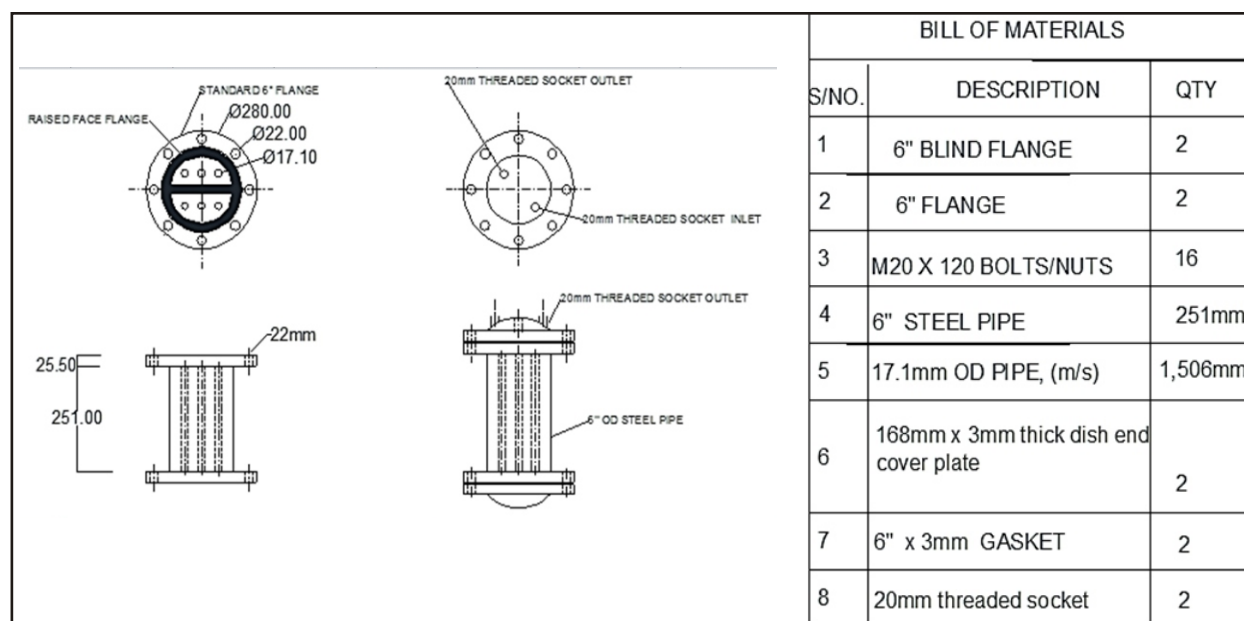


Figure 5. Orthographic Projections of the Multi-tubular Fixed Bed Reactor

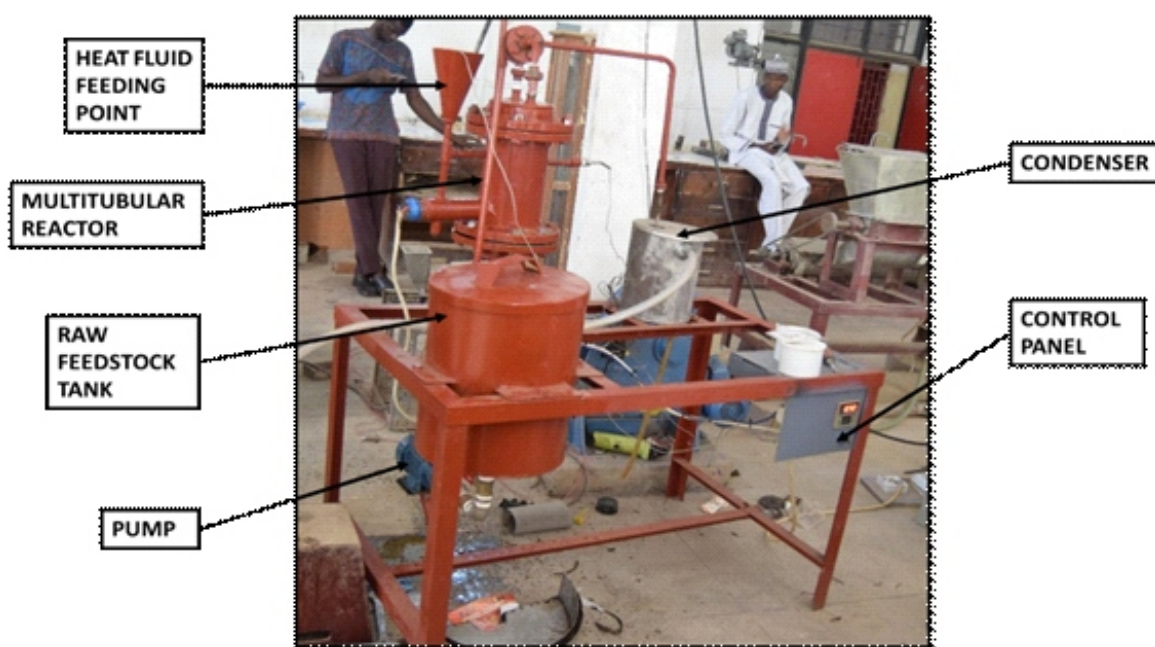


Figure 6. Multi-tubular reactor setup

The snapshot of the pilot plant we fabricated setup is presented in Figure 6. Different plant components were clearly explained, showing the reactor, pumps, raw material tank, condensing unit, heating fluid loading point, and control panel for the pilot plant operation.

3.4 Material Selection:

In selecting the suitable material for the fabrication of the reactor, glass, mild steel, and stainless steel were considered. Here, the materials were screened given the factors such as material fabricability, availability of the material, the appearance of the material, cost of the material, corrosion resistance of material and material strength.

Table 4. Material selection for the reactor fabrication [25], [27], [34][38]

Description	Variation of Materials		
	Glass	Mild Steel	Stainless Steel
<i>Material fabricability</i>	Easy to fabricate, but glass blowing machines are not readily available in Zaria/Kaduna unless in Minna	Easy to fabricate, because all welders can weld it.	Challenging to fabricate because not all welders can weld it.
<i>Availability of the material</i>	Readily available	Readily available	Readily available
<i>The appearance of the material</i>	It looks more excellent than all other materials for class demonstration because it is transparent, and students will be able to see what is happening inside the reactor.	It will not look nice because students will not be able to see what is happening inside the reactor unless disassembled. It can easily be eroded.	It will not look nice because students will not be able to see what is happening inside the reactor unless disassembled. But it will be better than mild steel because it has a better shining surface and is corrosion-resistant.
<i>Cost of the material</i>	Cheaper	Cheapest	Expensive
<i>The corrosion resistance of the material</i>	Good corrosion resistance	poor corrosion resistance	Good corrosion resistance
<i>Material strength</i>	It can have good strength, but it is highly brittle.	Good strength	Good strength

Mild steel was selected as a result of the availability, cost, strength, and fabricability of the material for the reactor [25], [27], [34], [35].

3.5 Start-up and Shutdown:

3.5.1 Start-up procedure:

The start-up procedure for the plant set-up is presented in the order below:

1. The Heating Oil is heated to about 250°C and then released into the Shell of the Reactor (MTFBR) by gravity. Point 5, the exit point on the Shell, remains closed. Ensure the heating is controlled by taking the temperature measurement intermittently.
2. Leave the heated oil for some time in the Shell and allow it to heat the tube of reactant to 200°C.
3. Release the Isopropanol from the Raw Material Tank (U-RT) and allow it to flow into reactor tubes.
4. The reaction occurs in the Reactor Tubes over the Solid Catalyst at stated operating conditions, and the products exit the tube as vapors, which are cooled by the condenser.
5. Collect the Acetone produced in the Product

Tank (U-PT).

3.5.2 Shut-down procedure

The shut-down procedure for the plant set-up is presented in the below order:

1. Stop the raw material from flowing into the Reactor Tube.
2. Allow the remains of the reactant inside the reactor to react and exit as products.
3. Stop the Oil flow into the reactor's Shell (MTFBR).
4. Allow the rest of the Oil exiting the Reactor Shell to drain completely.
5. Allow the acetone product to continue dropping until the Product Tank receives the final drop (U-PT).

3.6 Process Cost Analysis: Estimation of Total Capital and Cost of Operation

The cost analysis of the project was presented, reflecting the total capital and operation cost computation indicating the financial demands and benefits of the project are showcased here. The result obtained from the collection of the assets required to set up the process plant is presented in Table 5, including the project's total capital cost.

Table 5. Total capital cost and cost of operation

Capital Cost Descriptions	Amount (NGN)	Operation Cost Descriptions	Amount (NGN)
Equipment:		Catalyst materials:	
Raw material tank	5,000.00	Copper nitrate	6,500.00
Reactor	67,000.00	Silicon oxide	7,500.00
Condenser	15,000.00		
Product tank	5,000.00		
Pump	20,000.00	Reagent for acetone test:	
Pipe & fitting	25,000.00	Sodium hydrogen sulfate	Not accounted
		Fehling solution	
		Iodine solution	
		Sodium hydroxide	
Installation	20,000.00	Raw materials:	12,500
		Isopropanol	
Temperature Controller and indicators	16,000.00	Heating fluid/oil:	25,000
		Engine oil	
Support skid	28,000.00	Coolant/cooling materials:	1,000
		Ice-block	
Transport	10,000.00	Transport	10,000
Total capital Cost	211,000.00	Total operating cost	62,500.00

The total cost of operating the manufacturing or production stage of the plant was computed via the use of the cost of the operational resources required in the production of acetone from the isopropanol was presented in Table 5.

Findings from the cost analysis indicate that a total of 211 thousand nairas (USD 512.76) were deployed in the building, fabrication, installation, and transport of the equipment. A sum of 62 thousand and five hundred nairas (USD 151.88) was estimated as the required cost of running the pilot plant operation. The running (or operation) cost was relatively low and affordable for average schools in low-income countries relative to the cost of importing the ready-made version of the setup from abroad.

4. Conclusions:

A reactor was successfully fabricated using the dimensions specified in Table 5.1. The total capital invested in this design and fabrication amounted to NGN 211,000 (USD 512.76), while the operating cost was estimated to be NGN 62,500 (USD 151.88). The cost analysis indicates the running of the plant

for teaching and research in chemical engineering science, especially in low-income (developing) countries. The design specification provided in this study can be used by relevant chemical engineering departments where the modern facility is challenging to afford or access in their colleges and could be used in fabricating the local multi-tubular reactor setup, which would aid in facilitating the training of process engineers, especially in developing nations like Nigeria. That is, it would go a long way to promote the teaching of reactor design, reaction kinetics, and heterogeneous catalysis in schools. Moreover, the influence of heating fluid properties on the kinetics of reactions in the multitubular reactor can be considered in further study, and the factors to consider in selecting a suitable heating fluid to meet the heating demand of a particular chemical reaction in a specific study. Other studies can explore the economic potential of scaling the pilot design of the process studied in this report into a commercial scale in low-income nations.

Conflict of Interest

The authors declare no conflict of interest.

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References:

1. W. Allen, H. J. M. Bowen, L. E. Sutton, and O. Bastiansen, "The molecular structure of acetone," *Transactions of the Faraday Society*, vol. 48, no. 0, pp. 991995, Jan. 1952, doi: 10.1039/TF9524800991.
2. S. Sifniades, A. B. Levy, and H. Bahl, "Acetone," *Ullmann's Encyclopedia of Industrial Chemistry*, Jan. 2010, doi: 10.1002/14356007.A01_079.PUB2.
3. M. C. Tohaneanu, V. Plesu, P. Iancu, G. Bumbac, A. E. B. Ruiz, and J. B. Ruiz, "Simulation and Process Integration of Clean Acetone Plant," *Chem Eng Trans*, vol. 39, no. Special Issue, pp. 469474, Aug. 2014, doi: 10.3303/CET1439079.
4. CEN, "Production of Phenol and Acetone From Oxidation of Cumene Starts," *Chemical Engineering News*, vol. 31, no. 22, pp. 22982307, 2010, doi: 10.1021/CEN-V031N022.P2298.
5. V. M. Zakoshansky, "The Cumene Process for PhenolAcetone Production," *Petroleum Chemistry 2007 47:4*, vol. 47, no. 4, pp. 307307, Jul. 2007, doi: 10.1134/S0965544107040135.
6. W. L. Luyben, "Design and Control of the Cumene Process," *Ind Eng Chem Res*, vol. 49, no. 2, pp. 719734, Jan. 2009, doi: 10.1021/IE9011535.
7. J. Fu *et al.*, "Direct Gas-Phase Oxidation of Propylene to Acetone in the Presence of H₂ and O₂ over Au/TS-1 Catalyst," *Curr Org Synth*, vol. 17, no. 8, pp. 685690, Jun. 2020, doi: 10.2174/1570179417666200615154837.
8. A. El-Aziz, A. Said, M. M. M. Abd El-Wahab, and M. N. Goda, "Selective synthesis of acetone from isopropyl alcohol over active and stable CuO-NiO nanocomposites at relatively low-temperature," *Egyptian Journal of Basic and Applied Sciences*, vol. 3, no. 4, pp. 357365, 2016, doi: 10.1016/j.ejbas.2016.08.004.
9. T. Oyegoke, "Engineering the Performance of Cobalt in the Dehydrogenation of Isopropanol into Acetone via the Use of Multiscale Approach," 2021. doi: 10.13140/RG.2.2.26264.06406.
10. ChemEngNews, "Production of Phenol and Acetone From Oxidation of Cumene Starts," *Chemical Engineering News*, vol. 31, no. 22, pp. 22982307, 2010, doi: 10.1021/CEN-V031N022.P2298.
11. M. K. Vovdenko, I. M. Gubaidulin, K. F. Koledina, and S. N. Koledin, "Isopropylbenzene oxidation reaction computer simulation," in *3rd International conference "Information Technology and Nanotechnology*, 2017, p. 23. Accessed: May 13, 2022. [Online]. Available: <http://ceur-ws.org/Vol-1966/paper5.pdf>
12. J. Fu *et al.*, "Direct Gas-Phase Oxidation of Propylene to Acetone in the Presence of H₂ and O₂ over Au/TS-1 Catalyst," *Curr Org Synth*, vol. 17, no. 8, pp. 685690, Jun. 2020, doi: 10.2174/1570179417666200615154837.
13. W. L. Luyben, "Design and Control of the Acetone Process via Dehydrogenation of 2-Propanol," *Ind Eng Chem Res*, vol. 50, no. 3, pp. 12061218, Feb. 2010, doi: 10.1021/IE901923A.
14. J. M. Amezcua-Ortiz, H. Alcocer-Garcia, G. Contreras-Zarazua, J. Fontalvo, and J. G. Segovia-Hernandez, "Sustainable Process Design for Acetone Purification Produced via Dehydrogenation of 2-Propanol," *Ind Eng Chem Res*, vol. 61, no. 10, pp. 36603671, Mar. 2022, doi: 10.1021/ACS.IECR.1C04321.
15. B. Kökdemir and N. Acaraly, "A novel study on CHEMCAD simulation of isopropyl alcohol dehydrogenation process development," *Journal of the Indian Chemical Society*, vol. 98, no. 3, Mar. 2021, doi: 10.1016/J.JICS.2021.100035.
16. S. S. Lokras, P. K. Deshpande, and N. R. Kuloor, "Catalytic Dehydrogenation Of 2-Propanol To Acetone," *Industrial and*

- Engineering Chemistry Process Design and Development*, vol. 9, no. 2, pp. 293297, 1970, doi: 10.1021/I260034A022.
17. "US4472593A - Conversion of isopropyl alcohol to acetone - Google Patents." <https://patents.google.com/patent/US4472593A/en> (accessed Sep. 26, 2022).
 18. Lynn H. Slaugh, Galeon W. Schoenthal, and James D. Richardson, "Conversion of isopropyl alcohol to acetone," Dec. 09, 1982
 19. A. E.-A. A. Said, M. M. M. Abd El-Wahab, and M. N. Goda, "Selective synthesis of acetone from isopropyl alcohol over active and stable CuONiO nanocomposites at relatively low-temperature," *Egyptian Journal of Basic and Applied Sciences*, vol. 3, no. 4, pp. 357365, Dec. 2016, doi: 10.1016/J.EJBAS.2016.08.004.
 20. A. Balouch, A. Ali Umar, A. A. Shah, M. Mat Salleh, and M. Oyama, "Efficient heterogeneous catalytic hydrogenation of acetone to isopropanol on semihollow and porous palladium nanocatalyst," *ACS Appl Mater Interfaces*, vol. 5, no. 19, pp. 98439849, Oct. 2013, doi: 10.1021/AM403087M/ASSET/IMAGES/MEDIUM/AM-2013-03087M_0008.GIF.
 21. K. D. Ramachandriya *et al.*, "Reduction of acetone to isopropanol using producer gas fermenting microbes," *Biotechnol Bioeng*, vol. 108, no. 10, pp. 23302338, Oct. 2011, doi: 10.1002/BIT.23203.
 22. T. Jojima, M. Inui, and H. Yukawa, "Production of isopropanol by metabolically engineered *Escherichia coli*," *Applied Microbiology and Biotechnology* 2007 77:6, vol. 77, no. 6, pp. 12191224, Jan. 2008, doi: 10.1007/S00253-007-1246-8.
 23. V. P. Kudesia and S. K. Mukherjee, "Kinetics of Reaction of Isopropyl Alcohol with Aqueous Chlorine," *Indian Journal at Chemistry*, vol. 15, pp. 513516, 1977.
 24. R. W. Missen, C. A. Mims, and B. A. Saville, *Introduction to Chemical Reaction Engineering and Kinetics*. New York: John Wiley Sons, Inc, 1999.
 25. R. Turton, R. C. Bailie, W. B. Whiting, J. A. Shaeiwitz, and D. Bhattacharyya, *Analysis, Synthesis, and Design of Chemical Processes Fourth Edition*, 4th ed. New York: Prentice Hall, 2012.
 26. J. M. Coulson, J. F. Richardson, and R. K. Sinnott, *Chemical engineering. Vol.6, Chemical engineering design*, 4th ed., vol. 6. Elsevier Butterworth-Heinemann, 2006.
 27. R K Sinnott, J M Coulson, and J F Richardson, *Coulson & Richardson's chemical engineering: Chemical engineering design*, vol. 6. Elsevier Butterworth-Heinemann, 2005. Accessed: Jul. 26, 2021. [Online]. Available: <https://lib.fr/book/2359579/d933fc?id=2359579&secret=d933fc>
 28. R. Peña-Alonso, F. Rubio, J. Rubio, and J. L. Oteo, "Study of the hydrolysis and condensation of α -Aminopropyltriethoxysilane by FT-IR spectroscopy," *Journal of Materials Science* 2006 42:2, vol. 42, no. 2, pp. 595603, Nov. 2006, doi: 10.1007/S10853-006-1138-9.
 29. Y. Ye *et al.*, "Silica-Supported Copper (II) Oxide Cluster via Ball Milling Method for Catalytic Combustion of Ethyl Acetate," *Catalysts* 2022, Vol. 12, Page 497, vol. 12, no. 5, p. 497, Apr. 2022, doi: 10.3390/CATAL12050497.
 30. O. O. Fasanya, A. Y. Atta, M. T. Z. Myint, J. Dutta, and B. Y. Jibril, "Effects of synthesis methods on performance of CuZn/MCM-41 catalysts in methanol steam reforming," *Int J Hydrogen Energy*, vol. 46, no. 5, pp. 35393553, Jan. 2021, doi: 10.1016/J.IJHYDENE.2020.10.181.
 31. H. Li *et al.*, "Regulation of Cu Species in CuO/SiO₂ and Its Structural Evolution in Ethynylation Reaction," *Nanomaterials*, vol. 9, no. 6, p. 842, Jun. 2019, doi: 10.3390/NANO9060842.
 32. J. J. Reinoso, J. J. Romero, P. Jaquotot, M. A. Bengochea, and J. F. Fernández, "Copper based hydrophobic ceramic nanocoating," *J Eur Ceram Soc*, vol. 32, no. 2, pp. 277282, Feb. 2012, doi:

- 10.1016/J.JEURCERAMSOC.2011.08.013.
33. R. Turton, R. Bailie, W. Whiting, J. Shaeiwitz, and D. Bhattacharyya, *Synthesis and Optimization of Chemical Processes*, 4th ed. New York: Prentice Hall, 2012.
34. MSDSonline, "Isopropyl Alcohol Safety Tips from MSDSonline - VelocityEHS," *MSDSonline*, Jun. 26, 2021. <https://www.msdsonline.com/2015/02/20/isopropyl-alcohol-safety-tips-from-msdsonline/> (accessed Apr. 24, 2022).
35. MSDS, "MSDS Number A0446 Effective Date Supersedes ACETONE 1 Product Identification," Jun. 26, 2021. <https://www.coursehero.com/file/54273201/ACETONE-A0446pdf/> (accessed Apr. 24, 2022).
36. A. Jahan, M. Y. Ismail, S. M. Sapuan, and F. Mustapha, "Material screening and choosing methods - A review," *Mater Des*, vol. 31, no. 2, pp. 696705, Feb. 2010, doi: 10.1016/j.matdes.2009.08.013.
37. E. Y. Salawu, O. O. Ajayi, A. O. Inegbenebor, S. Akinlabi, S. A. Afolalu, and A. Aroyinbo, "Material Selection and Processing Techniques: Crucial Factors for Gear Engineering," *IOP Conf Ser Mater Sci Eng*, vol. 1107, no. 1, p. 012114, Apr. 2021, doi: 10.1088/1757-899X/1107/1/012114.
38. A. A. A. Rahim, S. N. Musa, S. Ramesh, and M. K. Lim, "A systematic review on material selection methods," *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications*, vol. 234, no. 7, pp. 10321059, May 2020, doi: 10.1177/1464420720916765.