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Concentrated Nitric Acid Production by Recycling and Rectification: Optimistic Approach for Better Yield

Bhanu Pratap Singh[1], Ishty Malhotra[2]
[1]*(School of Bio-Chemical Engineering, Indian Institute of Technology (BHU), Varanasi, India)*
[2]*(Department of Chemical Engineering and Technology, Indian Institute of Technology (BHU), Varanasi, India)*

Abstract: ***Nitric acid is considered as one of top world's producing chemicals, having uses in variety of sources and products. Utilization includes in manufacture of fertilizers, polyurethanes, nylon, polyamides filaments, as propellant for rockets in aerospace industries, explosives like trinitrotoluene, nitro-glycerine and some more. Its production and concentration involve importance only when carried out using the optimization techniques. Such a method is utilized here. Conventional rectification method alone mostly used to separate and purify chemical mixtures but has disadvantage of being an energy-intensive unit operation. In order to control the processing and yield of plant, the material, energy balances become fundamental. This paper discusses production of 130 metric tons concentrated nitric acid using recycling and rectification, serving an alternatively suitable approach with overall plant flow sheet under the assumption of steady state. The rectification module is intended to be utilized at the last phase of creating the predetermined acids. During the planning of this module, endeavours were made to guarantee measure proficiency to satisfy the prerequisites on manufacturability for segments, units and the entire establishment. The detailed description of process is explained which includes material and energy balance on every component using the chemical reactions involved in the equipment during component balancing. This approach helps to achieve to control the desired yield of product as well as can become the basics for further pilot plant calculations.***

Keywords: optimization techniques, concentrated nitric acid, recycling, rectification, material energy balance, flowsheet

I. INTRODUCTION

With the quickly developing situation of substance businesses, the most development popular has originated from the creation of polyurethanes, strands and alkali nitrate-based explosives [1]. The normal synthetic utilized in these enterprises is nitric acid [2]. The dismal to yellow or red fluid which once in a while exhaust as ruddy earthy colored fumes with choking out smell is utilized in huge territories. The creation of nitric acid includes the significant difficulties and thus required a total examined measure so as to confront the issues that must be tended to by humankind.

Nitric acid is generally called as solid (water fortis) by medieval times and is considered as solid inorganic acid which is delivered in enormous volumes [3]-[4]. It is utilized in the creation of different synthetics yet generally assumed function in manures industry [5]-[6]. Other than this, it is additionally utilized in arrangement of farming synthetic compounds, consumption inhibitors, intermediates, water treatment items, individual consideration items, hostile to scaling specialists, oxidizing operators and some more [7]-[8]. In any cycle activity; material amounts as they go through handling tasks can be portrayed by material adjusts. Such adjusts are explanations on the preservation of mass. On the comparable line, energy amounts can likewise be depicted by energy adjusts, which are explanations on the protection of energy [9]. In the event that there is no gathering, what goes into a cycle must come out. This is valid for cluster activity and furthermore similarly valid for the consistent activity throughout any picked time stretch.

Material and energy adjust are significant in a modern cycle. Material adjusts are essential to the control of preparing, especially in the control of yields of the items [10]. The primary material balance is resolved in the exploratory phases of another cycle, improved during pilot plant tests when the cycle is being arranged and tried, looked at when the plant is appointed and afterward refined and kept up as a control instrument as creation proceeds [11].

On the off chance that there is any adjustment simultaneously, the material adjusts should be resolved once more. In this paper, the material and energy balance has been performed upon the nitric acid concentration plant under the steady state that involves the detailed description of plant process followed by the material and energy balance using the block diagrams.

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II. NITRIC ACID CONCENTRATION PROCESS

Various processes take place during the formation of nitric acid as shown in various studies [12]. Nitric Acid Concentration measure (NAC) measures utilized extractive refining to think frail acid up to 99 wt. %. A drying out operator, for example, sulfuric acid or magnesium nitrate is utilized to upgrade the instability of nitric acid so refining strategy can outperform the azeotropic grouping of nitric acid [13]. Powerless acid and getting dried out operators are taken care of to a refining section. Water eliminated from the acid weaken the drying out specialist, which is taken out as a base stream and later gathered for reuse simultaneously. Super-azeotropic acid fumes pass to the lower part of correction area in which the acid is focused up to 99 wt. %. The solid nitric acid is consolidated overhead and a bit of acid is gotten back to segment as reflux. The 72% sulphuric leaving the lower part of the pinnacle is de-nitrated by steam stripping in a subsequent pinnacle, and the re-concentrated to 93% by contact with heaters gases in a drum concentrator.

A. Direct Strong Nitric Processes

The second way to deal with the convergence of nitric acid of late called the Direct Strong Nitric cycle, is an outgrowth of the environment climatic weight measure. At barometrical weight, the pace of oxidation of nitric oxide to the dioxide is incredibly moderate with the goal that brilliantly little dioxide is available when the gas arrives at the condenser after the alkali burner. The vast majority of the water framed can along these lines be dense in a barometrical plant shaping an answer with just 2-4% by weight nitric acid. Some portion of this acid is utilized as a cosmetic simultaneously, yet its majority has regularly been disposed of. The cycle gas leaving the condenser is reached by a moved nitric acid in an oxidation tower, switching the assimilation response by changing nitric oxide in addition to nitric acid over to nitrogen dioxide and water. The dioxide in the process gas is artificially assimilated into an azeotropic or more prominent quality acid. Air stripping and amendment of the subsequent steam produces solid acid of 98-99 wt. % quality.

B. Cycle Selection

We have chosen concentrated nitric acid cycle which is a direct solid acid cycle due for its after potential benefits over different cycles

1) Recycle circle and the exceptional amendment step are developments of this cycle. They take out the refrigeration oxygen and substance getting dried out specialists (model: - sulfuric acid on magnesium nitrate) needed by old style producing methods, and in this manner decline utilities and working costs extensively.
2) In nitric acid fixation measures, drum concentrators are utilized and electrostatic precipitators are given to wipe out acid from the vented gas. They have working issues and consequently contamination issues are face, though in CNA measure, barometrical outflows have been under 450 ppm in NOx with no decrease units.
3) This plant is completely robotized, just two administrators shifts are required. This cycle can likewise make concentrated or frail acid 50-60% in any extent, the item blend can be changed by changing the situation of two dividers. This course as a low speculation when contrasted with different courses.

III. DETAILED PROCESS DESCRIPTION

Liquid ammonia astounded and is blended in with sifted stream of air. The converter area comprises of 10-30 sheets of Pt/Rh alloy as 60-80 work wire gage stuffed in a layer inside the cylinder. Gas passes descending with speed intended to give contact season of about $2.5*10^{-4}$ sec in the impetus zone at 870^{o} C . The mix responds with Pt/Rh impetus at about 870^{o}C and climatic strain to shape nitric oxide and water. Item gases from reactor, containing 10-12% NO, are sent through warmth recuperation units, an extinguish unit for quick cooling to eliminate an enormous division of item heat and into the oxidizer safeguard framework. Response items go through the tail gas preheater, squander gas evaporator and cooler condenser to disseminate the warmth created. Air is added to change NO over to NO_2 at the most ideal low-temperature (40-50^{o}C) climate of the ingestion framework. At barometrical weight, the pace of oxidation of nitric acid to dioxide is incredibly moderate with the goal that generally little dioxide is available when the gas arrives at the condenser [14]. All the water present is consolidated and is depleted off as 2% by wt. nitric acid arrangement. Activity at climatic constrain assists with holding acid substance down. The hardware in the retention train possibly arrangement of certainty or strainer plate vertical pinnacles. The dry gas stream is preheated and set to lower part of oxidation tower. Here 50-60% acid from the assimilation part of the framework and enters the highest point of the section. As the gas and the fluid stream counter as of now, the acid oxidizes NO to NO_2.

$$NO + 2HNO_3 \rightarrow 3NO_2 + H_2O$$

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Weaken acid from the lower part of the oxidation unit is ship off assimilation segment of the root for fixation. The cycle gas leaving the highest point of oxidation tower is joined with air containing nitrogen oxide and afterward compacted. The compacted gas is cooled prior to entering the ingestion segment of the cycle. This segment really comprises of two segments, one that delivers the super azeotrope and different makes a frail acid [15]. The gases initially enter the super azeotrope section and are reached with azeotrope (68%) acid from the refining unit. Nitrogen dioxide is assimilated and a super azeotrope of 80% nitric acid is shaped. The super azeotrope soaked with nitrogen oxide is stripped with air, preheated and afterward took care of two refining section. The typical is a subject acid combination utilized for assimilation is taken of the lower part of refining section. The typical azeotropic acid is blend utilized for retention is removed the lower part of refining segment. Concentrated acid 98% is acquired at the top [16]. Since the cycle gas leaving the highest point of the super azeotropic assimilation section are as yet wealthy in nitrogen oxide. They are shipped off another ingestion unit. Here, low fixation acid from the oxidation tower enters in the centre. Nitric Acid which is around 50-60 % is taken out from the base. The current gas has centralization of NOx in the request for 300-400 ppm. That tail gas is preheated to about 450^{o} C by measure streams in the alkali oxidizer and the streams leaving the blower as shown in Figure: 1 [17] and Table: 1 explains the names of the unit operations used. The rectification module is intended to be utilized at the last phase of creating the predetermined acids. The rectification process were made to guarantee measure proficiency to satisfy the prerequisites on manufacturability for segments, units and the entire establishment.

A. Grouping of Nitric Acid To 95% Can Be Done by one of Several Process

1) *Consideration by Sulphuric Acid:* Rectification by 93% sulphuric acid in silicon iron mineral stoneware towers produces concentrated nitric acid and 70% sulphuric acid can be the dissipated to 93%.
2) *Concentration by Magnesium Nitrate:* Magnesium Nitrate arrangement containing 70-75% magnesium nitrate is taken care of to the nitrate drying out plate tower alongside weaken nitric acid from ingestion framework. The salt arrangement go about as an extractive refining specialist, eliminating water at 100oC or higher was permitting correction without azeotropic development [18]. Weaken magnesium nitrate arrangement is re-concentrated by dissipation.

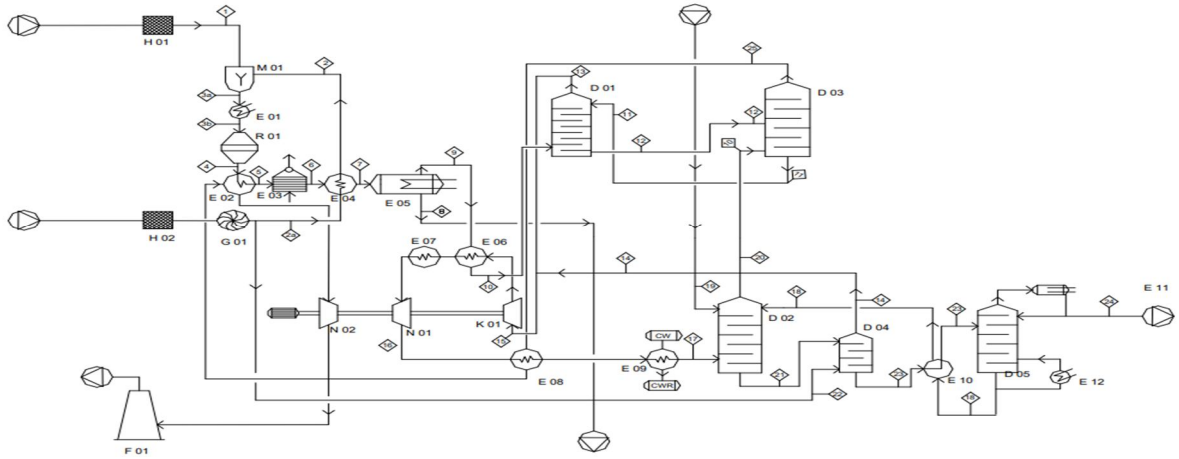

Figure 1: - Flowsheet for Production of Concentrated Nitric Acid using Recycling and Rectification

Table 1: - List of Unit Operations Used

H01 Ammonia Filter	E01 Heat Exchanger	E06 Heat Exchanger	E11 Condenser	D04 Bleaching Tower
H02 Air Filter	E02 Tail Gas Pre-Heater	E07 Heat Exchanger	E12 Reboiler	D05 Distillation Column
M01 Mixer	E03 Waste Heat Boiler	E08 Heat Exchanger	D01 Oxidation Tower	K01 Compressor
R01 Reactor	E 04 Air Pre-Heater	E09 Heat Exchanger	D02 Super Azeotrope Tower	N01 Expander
F01 Stack	E05 Cooler/Condenser	E10 Heat Exchanger	D03 Weak Acid Tower, (absorption tower)	N02 Motor

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B. Focal points of Magnesium Nitride Process

1) Operating cost about portion of those experienced with sulfuric acid fixation
2) Capital prerequisites are 70% just as extraordinary
3) Acid quality and yield is improved.

IV. MATERIAL BALANCE

Basis of 130 metric tons/day has been taken on 100% basis. In one hr, 5416.667 kg/hr acid is produced with a total solution of 5527.211 kg/hr including the water amount as 110.544 kg/hr. Since 1 kg mole ammonia is required to produce 1 kg mole nitric acid therefore, ammonia required is 269.841 kg/metric ton nitric acid or 1461.638 kg/hr or 85.978 kg mol/hr. Since, overall ammonia to air ratio is 1:10, therefore, air required is 859.787 kg mol/hr.

As discussed above, primary air will enter from ammonia oxidation reactor, hence, the stochiometric composition of gas is 15% by vol% of ammonia in air. However, this composition is in explosive range of ammonia-air mixture, so, atmospheric plants operate at 14%.

Therefore, primary air entering is 528.150 kg mol/hr, oxygen entering in reactor is 110.911 kg mol/hr and nitrogen entering in reactor is 417.239 kg mol/hr.

Taking material balance around individual component we get,

A. Reactor

Reactor (R01) showing the material balance (Figure 2) which involves the shown two reactions as

$$4NH_3 + 5H_2 \rightarrow 4NO + 6H_2O \ (98\%)$$
$$4NH_3 + 3O_2 \rightarrow 2N_2 + 6H_2O \ (2\%)$$

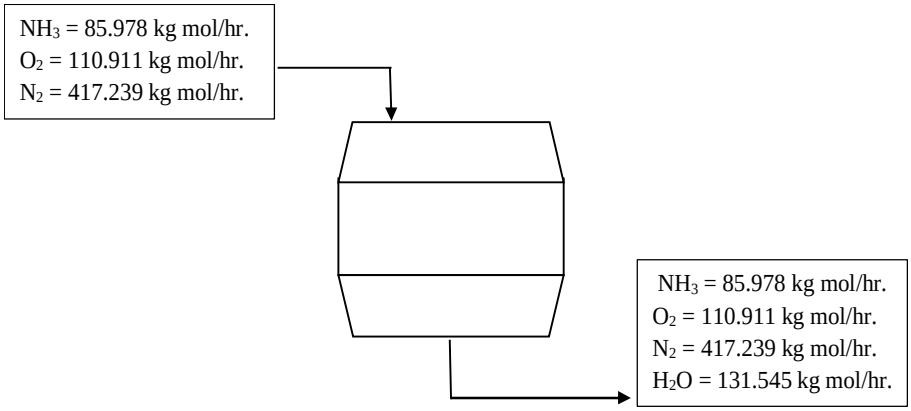

Figure 2: Material Balance on Reactor

B. Preheater And Bioler

Here, the reaction of 100% conversion takes place in tail gas pre-heater and in boiler(Figure: 3) (E-02 and E-03) which nitric oxide reacts with oxygen to gives nitrogen dioxide. Material balance is given as

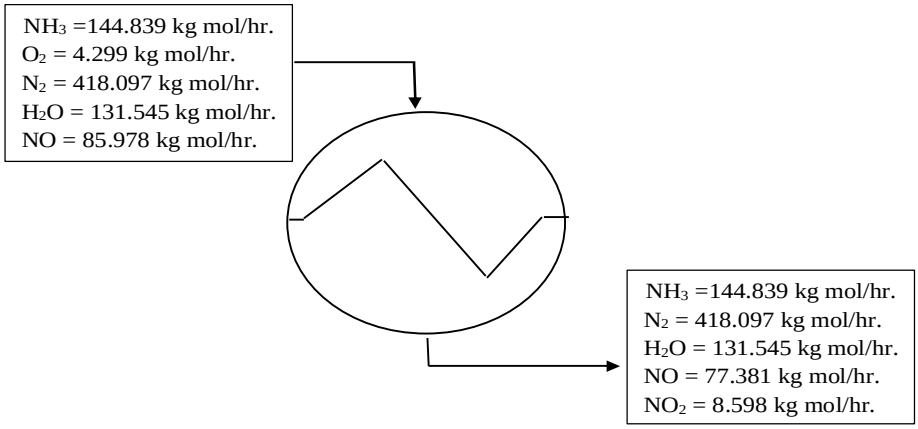

Figure 3: Material Balance on Preheater and Boiler

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C. Cooler/Condensor

Here, in cooler / condenser (Figure: 4) (E05), it is assumed that water will be drained as 2% of nitric acid solution by the reaction given below. For the reaction to proceed, it is also assumed that by condensation of water, nitric acid produced is 0.85 kg mol/hr. Material Balance is shown as

$$3NO2 + H2O \rightarrow 2HNO3 + NO$$

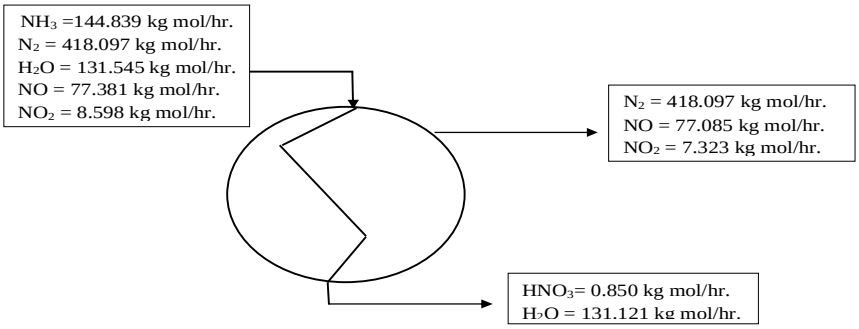

Figure 4: Material Balance on Cooler and Condenser

D. Oxidation Tower

Oxidation of nitric oxide takes place in oxidation tower (D01) with 90% conversion leads to the formation of water and nitric dioxide. Material balance (Figure: 5) is shown as: -

$$NO + 2HNO_3 \rightarrow 3NO_2 + H_2O\ (90\%)$$

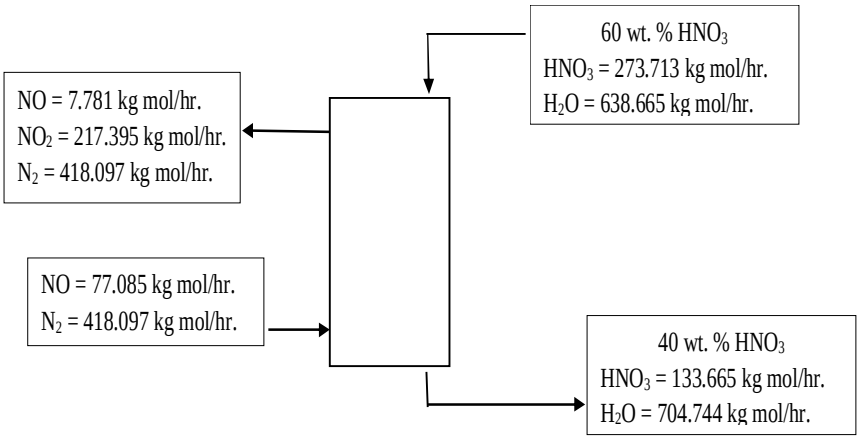

Figure 5: Material Balance on Oxidation Tower

E. Compressor

100% conversion takes place of nitric oxide which leads the formation of nitrogen dioxide in compressor (Figure: 6) (K01).

$$2NO + O_2 \rightarrow 2NO_2$$

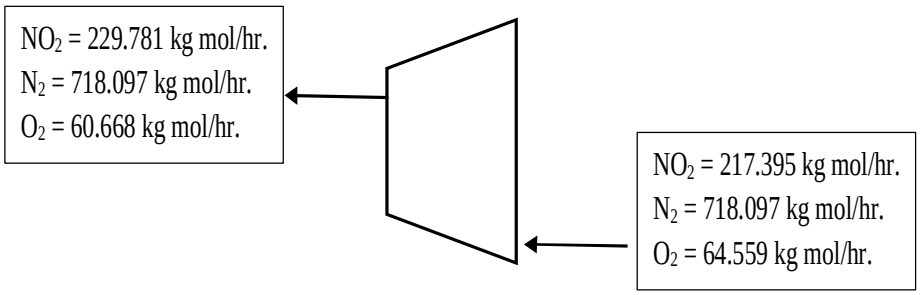

Figure 6: Material Balance on Compressor

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F. Distillation Column

Since, the initial acid solution taken was 5527.210 kg mol/hr. Hence, balancing over the distillation column (Figure: 7) (D05) as

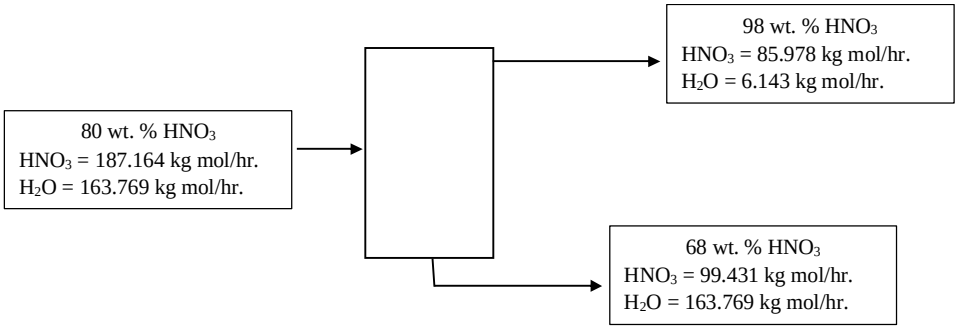

Figure 7: Material Balance on Distillation Column

G. Superazeotropic Column

Reaction $3NO_2 + H_2O \rightarrow 2HNO_3 + NO$ takes place in the column (D02). 4-5% NO2 produced goes towards the bleaching column (Figure: 8) for further process.

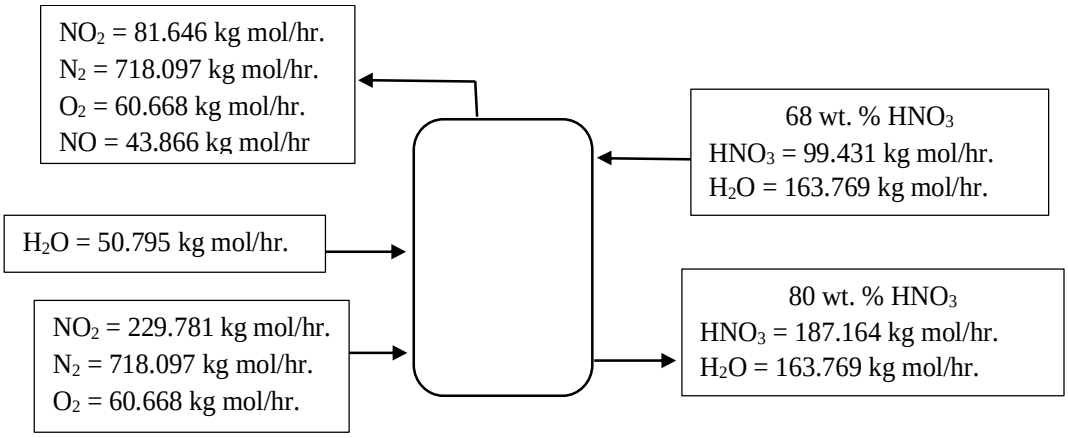

Figure 8: Material Balance on Super-azeotropic Column

H. Absoprtion Column

Following two reactions takes place in absorption column (D03) (Figure :9), on which the material balance has been applied at the steady state.

$$4NO + 3O_2 + 2H_2O \rightarrow 4HNO_3$$
$$4NO2 + O_2 + 2H_2O \rightarrow 4HNO_3$$

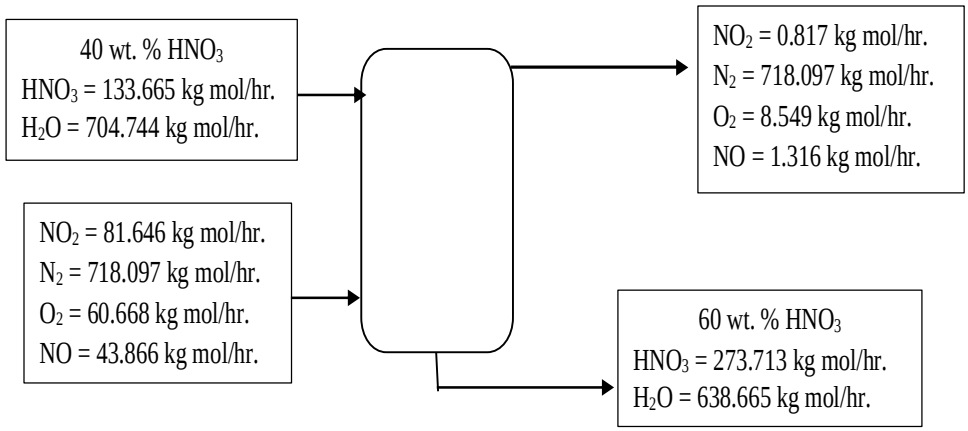

Figure 9: Material Balance on Absorption Column

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I. Bleaching Column

Since, the total air required is 859.787 kg mol/hr and the primary air inlet is 528.150 kg mol/hr (D04). Hereby, the balance (Figure :10) becomes,

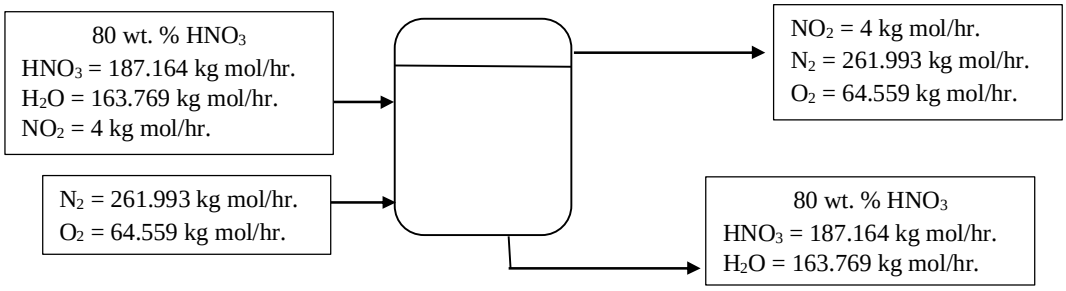

Figure 10: Material Balance on Bleaching Column

J. Overall Material Balance

Since, the material balance (Figure :11) has been taken up at steady state which clearly states that input = output of production. Hereby, the overall material balance is,

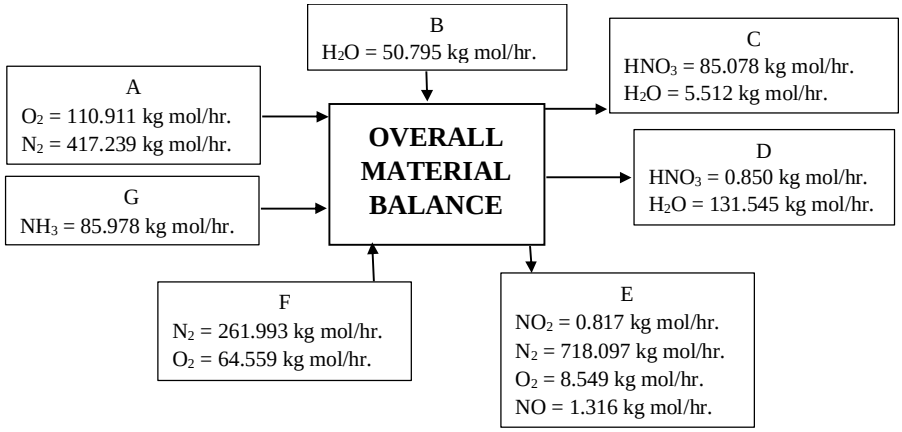

Figure 11: Overall Material Balance on Plant

V. ENERGY BALANCE

With the growing period of time, the expanding cost of energy has made the ventures analyse methods for decreasing energy utilization in preparing. Energy adjusts are utilized in the assessment of the different phases of a cycle, over the entire cycle and in any event, reaching out over the complete creation framework from the crude material to the completed item. Energy balances over the various equipment's takes place in the plant and assumptions being taken as steady state and specific heat values in the following approximate calculations. For the reactor, the two following reactions takes place as,

$$4NH_3 + 5O_2 \rightarrow 4\ NO + 6\ H_2O$$

$$4NH_3 + 3\ O_2 \rightarrow 2\ N_2 + 6\ H_2O$$

The total heat of the reactor is -1258092.885 kcal whereas, the total heat of reaction becomes -5173530.620 kcal/hr. Moving towards the two-stage compressor, the pressure difference has been taken as 9 atm, with the efficiency of 78%. The temperature of compressor at stage one is taken from 48^{O}C to 477.05^{O}C, whereas after the passage of intercoolers, the temperature changed from 50^{O}C to 185.45^{O}C. Similarly, the inlet temperature of oxidation tower is 50^{O}C, and the total enthalpy of reactants is taken as 450632.78 kcal and outlet temperature to be 46.5^{O}C.

Since, absorption column contains the 15 different coils, the total heat transferred through the coils is $2.42 * 10^{7}$ kJ/hr and total heat evolved by the two reactions comes out as $-2.58 * 10^{7}$ kJ/hr. Similarly heat exchanger, contains cold and hot fluid, process gases coming from cooler/condenser contains inlet temperature of 25^{O}C and outlet temperature of 50^{O}C, whereas, the hot fluid contains inlet temperature of 75^{o}C. Total heat of cold fluid is 95002.85 kcal/hr and for hot fluid is 87202.65 kcal/hr. The energy balance on partial condenser contains the total heat load of 2495200.985 Btu/hr with the temperature limit of 25^{o}C to 95^{o}C. Hereby, the energy balance on all the major equipment takes place.

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VI. CONCLUSION

Nitric acid concentration process has been shown using the recycling and rectification method, in order to produce nitric acid by optimization method. Material balance has been done for the production of 130 metric tons of nitric acid and energy balance described above with the help of block diagrams and the chemical reactions associated with respected individual sections, provides the optimization and idea of consumption of energy during the plant process. Such type of study is important in order to understand the average production scenario in any plant. They help the understudy in picturing exactly what is occurring in quantitative experiments. Clearly, the more muddled the issue, the more valuable these helps become. In similar fashion, we can thus conclude that recycling along with rectification as the energy saving method that was found to be around 10~15% for general recycles in production of concentrated nitric acid which we will further present in our next paper.

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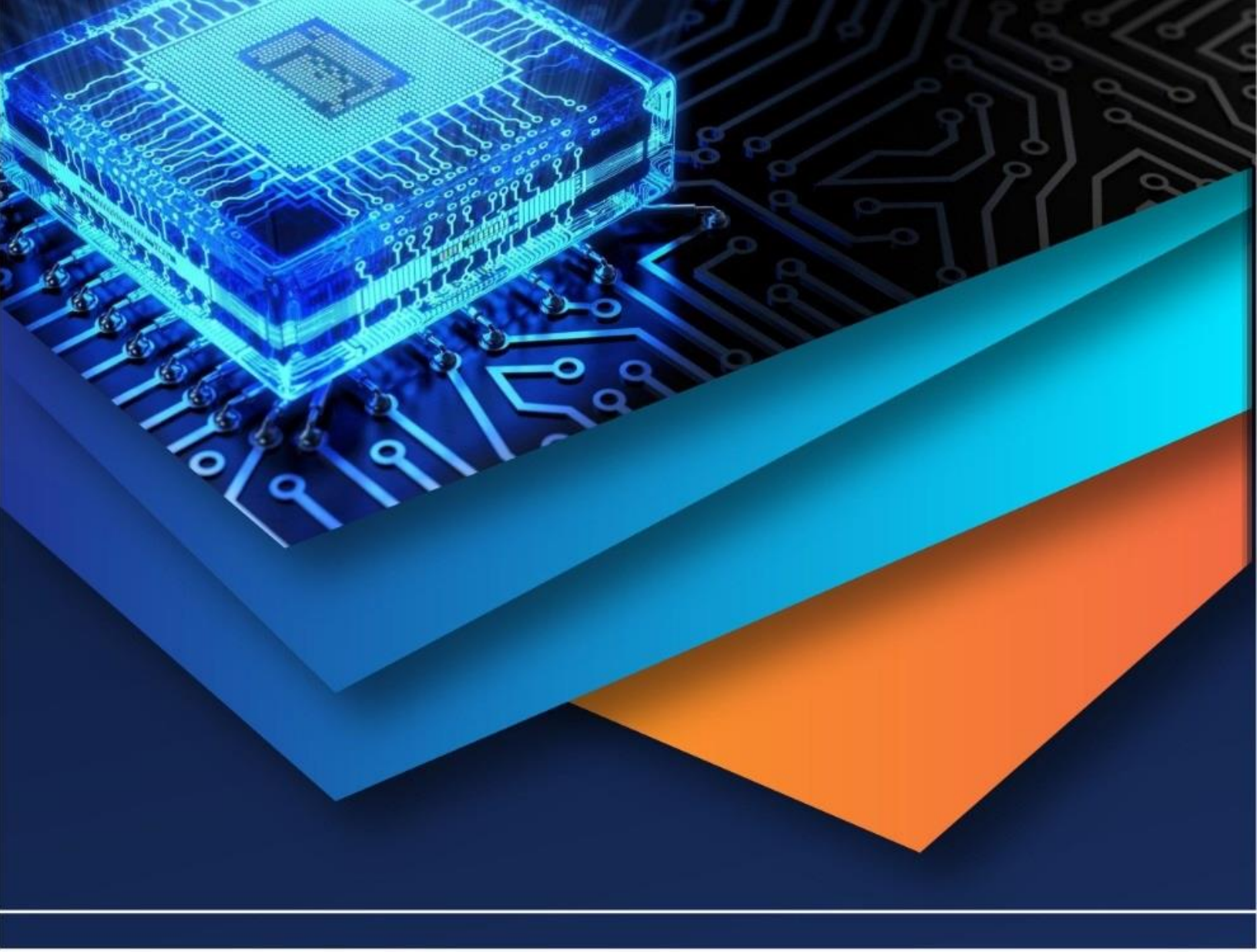

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