

# Some Investigation on Reflux Divider for Analysis of Reflux Ratio

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## Abstract

*This process is used to separate two or several solvents from a mixture. In fractional distillation process, a crude or a mixture of solvents is heated up to boiling temperature. The fractional distillation process is carried out when the difference in boiling point temperatures of different solvents is below 250C. For this operation, separate tanks are required for storing different solvents also it involves manual labor to keep an eye on the purity of product required. So, for eliminating this problem, we have designed a distillation column which will be operating on a suitable automatic mechanism. The distillation column consists of two ports for carrying reflux and collection operations. These two operations operated automatically by an electric timer circuit, which is work on timings provided by maintaining the purity of the product. By using reflux divider, time required for completing the distillation process is reduced as well as the percentage of purity also increase as there is no human errors. Hence, as the reduced time required, the production rate of the plant increased simultaneously.*

*Keywords: Automation, Fractional Distillation, Solenoid operated valve, Reflux ratio*

## 1. Introduction

In the traditional fractional distillation process, inlet & outlet valves of the distillation column were operated by workers only. The time required for completing the distillation process depends upon the separation of solvents, which is controlled by workers. This accuracy of separation is fully dependent on attention, skills & experience of the worker. As the overall process is under vacuum the conventional magnet powered & pneumatic reflux divider experienced the problem of jamming of dividing funnel into one of the collection or reflux ports. Also, the use of a pneumatic system was hazardous as the storage tank is to be placed near the reactors as well as the cost of the plant increased. In the case of magnetic reflux divider, the effect of the magnetic field was dependent upon the temperature of the solvent. As the temperature of the solvent increases, the magnetic effect decreases and hence, the performance of the reflux divider is affected.

The need for fractional distillation arises when the boiling temperature difference between two solvents is less than 250C. i.e. when the temperature of the distillation column reaches the boiling point of such solvents, it is possible that vapours of both the solvents may vaporize at the same time. Hence, to avoid such a situation, fractions of the solvents are taken otherwise simple distillation can be used. Therefore, fractional distillation must be used to separate the components by repeated vaporization-condensation cycles within a packed fractionating column. The application of distillation can roughly be divided into

four groups: laboratory scale, industrial distillation, the distillation of herbs for perfumery and medicinal (herbal distillate), and food processing.

The distillation process appears to have been utilized by the earliest experimentalists. Aristotle (384–322BC) mentioned that pure water is made by the evaporation of seawater. Pliny the Elder (ad 23–79) described a primitive method of condensation in which the oil obtained by heating rosin is collected on wool placed in the upper part of an apparatus known as a still. Bamberg et al. (2000) stated that the methodology of rapid machine design attempts to shorten the design-to-manufacture time of production equipment by using advanced engineering tools such as Computer-Aided Design systems (CAD) and Finite Element Analysis

(FEA) during the conceptual design phase. It is hypothesized that by identifying the best of all available design concepts, overall development time can be shortened [1].

Further time savings result from building machine components out of fabricated structures instead of casts. This eliminates the need for making moulds and other specialized tooling systems and provides a high degree of flexibility in terms of changing the design and making modifications to design specifications. Most methods of distillation used by industry and in laboratory research are variations of simple distillation. This basic operation requires the use of a still or retort in which a liquid is heated, a condenser to cool the vapour, and a receiver to collect the distillate. This simple apparatus is entirely satisfactory for the purification of a liquid containing non-volatile material and is adequate for separating liquids from widely divergent boiling points. For laboratory use, the apparatus is commonly made of glass and connected with corks, rubber bungs, or ground-glass joints. For industrial applications, larger equipment of metal or ceramic is employed [2].

A method called fractional distillation, or differential distillation has been developed for certain applications, such as petroleum refining, because simple distillation is not efficient for separating liquids whose boiling points lie close to one another. In this operation, the vapours from distillation are repeatedly condensed and vaporized in an insulated vertical column. Especially important in this connection are the still heads, fractionating columns, and condensers that permit the return of some of the condensed vapour toward the still. The objective is to achieve the closest possible contact between rising vapour and descending liquid to allow only the most volatile material to proceed in the form of vapour to the receiver while returning the less volatile material as liquid toward the still. The purification of the more volatile component by contact between such counter current streams of vapour and liquid is referred to as rectification or enrichment [3,4].

## **2. Design of System**

The design consists of an application of a scientific principle, technical information, and imagination along with the working ability of the machine for development of a new machine to perform a specific function with maximum economy and efficiency. Hence a careful design approach has to be adopted. The total design work has been split up into two parts as system design and mechanical design

### *2.1. System Design*

System design mainly concerns the various physical constraint and ergonomics, space requirements, the arrangement of various components on the mainframe at the system, man plus machine interaction number of controls, working environment of the machine, chances of failure. Safety measures to be provided ease of maintenance, the scope of improvement, the total weight of the machine, and a lot more.

### *2.2. Mechanical Design*

In mechanical design the components are listed down and stored based on the procurement, design in two categories namely, 1] Design Parts 2] Part to be purchased

1] Design Parts For design part detached design is done and distinction thus obtained is compared to next highest dimension which available in the market. This amplifies the assembly as well as postproduction servicing work with the better quality of parts of the system. The various tolerances of work are specified. The process chart is prepared and post on to the manufacturing stage. 22`

2] Parts to be purchased The parts which are to be purchased are selected from various catalogs and specified so that anybody can purchase the same from the retail shop with the given specification

### *2.3. Paramours for design*

System selection based on physical constraints while selecting any machine; it must be checked whether it is going to be used on a large scale or small-scale industry. So, space is a major constraint. The system is to be very compact so that it can be adjusted to the corner of the room. The mechanical design has direct norms with the system design. Hence the foremost job is to be controlled the physical parameter, so that distinctions obtained after mechanical design can be well fitted into that. Design steps: a. Design of reflux column b. Design of the timer circuit

**2.4. Design of Reflux Column**

While designing the reflux column, the basic dimensions of the equipment were design based on the discharge available at the condenser outlet.

The discharge available at the condenser outlet is 500ltr/hr.

Using the relation,

Discharge = Area x velocity  $Q=A \times V$  Eq.-----1

Where,

$Q = 500 \text{ LPH} = 500 \times 10^{-3} / 3600 = 1.389 \times 10^{-4} \text{ m}^3 / \text{sec}$

$A = \pi \times D^2 / 4 = \pi \times 0.0254^2 / 4 = 5.067 \times 10^{-4} \text{ m}^2$

$\therefore V = 0.281 \text{ m/sec}$

Selecting standard pipe diameter = 1" SS Pipe 23

Using empirical relation,

Flange diameter = 1.25 x diameter of pipe

Standard slip on MS flange diameter =1.25"

SS stub bend with O.D. = 35 mm & L = 102 mm

**2.5. Design of Receiver**

The receiver is meant to hold and separate solvents during the process. Hence while designing, we must consider the total volume of solvents to be separated.

We know that,  $Q=A \times V$

Where,

$Q = 1.389 \times 10^{-4} \text{ m}^3 / \text{sec}$   $V = 0.281 \text{ m/sec}$

$\therefore$ The dimensions of the receiver are as follow  $D = 320 \text{ mm}$  &  $L = 280 \text{ mm}$

**2.6. Design of Cut Piece Section**

The cut piece is meant to direct the flow of solvents either to reflux or for collection. The cut piece is fabricated at a distance of 70 mm from the base of the receiver and an angle of 35°.

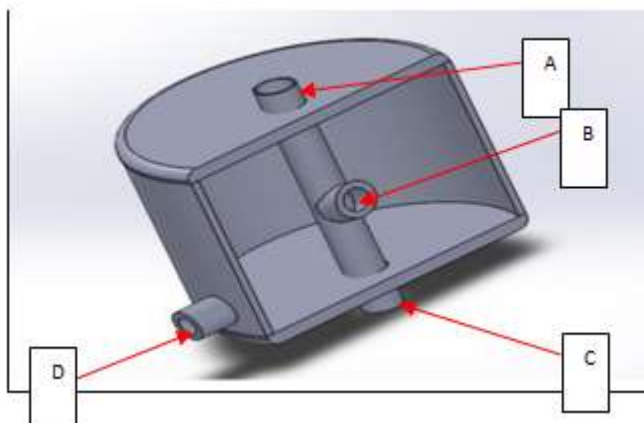


Fig. 1 Reflux Divider (Source: Solid works 2016)

A. Inlet port from condenser B. Reflux Divider port C. Reflux port to distillation column D. Collection port to collection vessel

The length of the cut piece is 50mm with a diameter of 1”

a. Design of T- section: The T section is used to direct the flow of solvents towards the solenoid valve. It is used to change the direction towards the lower receiver when the solenoid valve is under maintenance.

b. T- Section dimensions = 1.5” x 1” with the length of 145mm It is provided with a nozzle inside the T-section to increase the velocity of flow.

### 3. Working Methodology

First of all, the impure mixture is heated in a furnace with the help of an oil fired boiler. The heating is done until the temperature of the furnace reaches 180-190<sup>0</sup>C. This takes about 70-80 minutes of heating under a vacuum of 700mm of Hg As the temperature rises the low boiler, i.e. the solvents with low boiling temperature starts to vaporize. Due to temperature rise, the vapours rise and pass through the distillation column. In the distillation, column baffles are provided, which condenses water in the vapours, and only vapours rise and are passed through condenser. In the condenser the vapours are cooled by absorbing latent heat using cooling coils. This saturated liquid enters the receiver tank and starts accumulating it. When the solenoid valve is closed, this liquid flows towards the distillation column through the U bend. When the solenoid valve opens, the liquid flows into the collection port.

The above procedure is followed for 3-4 hrs. Until the desired quality of solvent is achieved during the starting phase the distillation column is full of impurities such as water when the temperature of the column is about 40-50<sup>0</sup>C. These water vapours are condensed and then passed through the collection port by opening the solenoid valve. (Fig.2)



Fig. 2 Actual Reflux divider in the distillation process.

Further when the temperature rises to 70<sup>0</sup>C the distillation column is enriched with butanol. These vapours are fractionated in reflux ratio 2:5 i.e. 2 minutes reflux and 5 min collection. Similarly when the distillation column attains the respective boiling temperatures of Xylene (90<sup>0</sup>C) and Octanol (110<sup>0</sup>C). Their vapours are refluxed and collected similarly. Finally only 2EHA vapours remain in the column which is the final product needed to obtain the required purity. The reflux ratio needs to be changed after certain intervals. During the early stage with high impurities the collection time is more than reflux. Time example, 2:8 as time passes, and vapours of the final product start enriching distillation column; there flux time is increased. Example, 8:2. In this way the whole fractional distillation process is carried out. The operation of reflux and collection port is controlled by a solenoid valve that operates on a timer switch, which can be pre-set as per requirements.

### 4. Results and Discussion

The test results show that the required purity of the final product, i.e.2EHA is attained after taking six online samples.

**Table 1 Influence of EP parameters on surface roughness**

Sample number	Component Name	Percentage in the 1 <sup>st</sup>	Percentage in the final
1	Water	0.85	0.00
2	Butanol	7.20	0.00
3	Xylene	1.65	0.24
4	Octanol	5.94	1.20
5	2EHA	<b>81.62</b>	<b>98.56</b>

Table1 shows that, the purity of 2EHA increased from 81.62% to 98.56% in 3 hours.

**5. Conclusions**

From this experiment, It is concluded that the distillation column first gets enriched with low boilers, i.e. the impurities with low boiling temperatures such as water vapours, butanol. The reflux ratio increased from 81.62% to 98.56% in 3 hours. Depending upon the reflux ratio, the timer On-time and OFF time is set which controls the ON/OFF position of the solenoid valve.

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