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Modelling, Simulation and Optimization of Fatty Acid Methyl Ester Reactive Distillation Process Using Aspen HYSYS

Samson Chukwufumnanya NWAMBUONWO[1], Abdulwahab GIWA[2]

[1]Chemical and Petroleum Engineering Department, College of Engineering, Afe Babalola University, KM. 8.5, Afe Babalola Way, 360231, Ado-Ekiti, Ekiti State, Nigeria

[2]Chemical and Petroleum Engineering Department, College of Engineering, Afe Babalola University, KM. 8.5, Afe Babalola Way, 360231, Ado-Ekiti, Ekiti State, Nigeria

Abstract: ***In this work, the modelling, simulation and optimization of a reactive distillation process used for the production of fatty acid methyl ester (FAME) has been carried out. The FAME considered was methyl palmitate, which was produced from an esterification reaction between palmitic acid and methanol. The reactive distillation column used for the process was set up in Aspen HYSYS environment using Distillation Column Sub-Flowsheet and the fluid package employed was Wilson model. The column had 17 stages, excluding the condenser and the reboiler, and it was divided into seven sections, viz, condenser section, rectifying section, upper feed section, reaction section, lower feed section, stripping section and reboiler section. Palmitic acid (fatty acid) entered the column through the upper feed section while methanol (alcohol) was fed at the lower feed section of the column. The developed model was simulated to convergence using Sparse Continuation Solver. Furthermore, the optimizer tool of Aspen HYSYS was used to obtain the optimum operating conditions of the process using three different algorithms (Box, Mixed and Sequential Quadratic Programming). The good convergence obtained from the simulation carried out on the developed Aspen HYSYS model of the reactive distillation process showed that Aspen HYSYS has been able to handle this process successfully. Furthermore, the achievement of the value of the objective function given by the optimization of the process when the estimated optimum values of reflux ratio, feed ratio and reboiler duty were used to run the model revealed that the optimum values obtained from Aspen HYSYS were theoretically valid. Therefore, it has been shown that the developed Aspen HYSYS model of this research work can be used to represent, simulate and optimize a FAME reactive distillation process successfully.***

Keywords: Fatty acid methyl ester, methyl palmitate, modelling and simulation, optimization, Aspen HYSYS.

1. Introduction

Reactive distillation (RD) process has been given special attention in the past two decades because of its potential for process intensification with certain types of chemical reactions (Popken *et a*l., 2001; Murat *et al*., 2003). It is a growing chemical unit operation that involves the integration of a reactor and a distillation column in one unit, i.e., it merges two different unit operations in a single piece of apparatus. In other words, reactive distillation involves simultaneous chemical reaction and multi-component distillation. The chemical reaction usually takes place in the liquid phase or at the surface of a solid catalyst in contact with the liquid phase (Seader *et al*., 2006).

The most important benefit of reactive distillation technology is a reduction in capital investment, because two unit operations can be carried out in the same equipment. Such integration leads to low costs of pumps, piping and instrumentation. For exothermic reaction, the reaction heat can be used for vaporization of liquid. This leads to savings of energy costs by the reduction of reboiler duties. Reactive distillation process is also advantageous when the reactor product is a mixture of species that can form several azeotropes with each other. Despite all these benefits, the combination of reaction and separation (distillation) is only possible if the conditions of both unit operations can be combined (Taylor and Krishna, 2000).

Reactive distillation can be applied to a variety of chemical reactions such as acetylation, aldol condensation, alkylation, amination, dehydration, esterification, etherification, hydrolysis, isomerization, oligomerization, transesterification, etc. For example, Giwa (2012) has applied it to a transesterification process used for producing n-butyl acetate. Giwa and Giwa (2012) have applied the process to optimize a transesterification reaction yielding methanol and n-butyl acetate. It (reactive distillation process) has been applied to production of isopropyl myristate by Giwa and Giwa (2013). Giwa *et al.* (2013) has applied it to carry out the simulation and economic analysis of ethyl acetate production. Also, Giwa (2013) has applied it to carry out the sensitivity analysis of ETBE production process. The esterification and/or transesterification reaction carried out in a reactive distillation column can be used to produce an important product referred to as fatty acid methyl ester (FAME).

Fatty acid methyl esters (FAMEs) are a type of fatty acid ester derived by transesterification of fats with methanol. They are used to produce detergents and biodiesel. Fatty acid esters are produced by vegetable oils and animal fats transesterification with short chain aliphatic alcohols. This process reduces significantly the vegetable oils viscosities without affecting its calorific power, thereby, allowing their use as fuel. Fatty acid methyl esters are typically produced by an alkali-catalyzed reaction between fats and methanol in the presence of a base such as sodium hydroxide or sodium

methoxide. The physical properties of fatty acid esters are closer to fossil diesel fuel than pure vegetable oils (Nwambuonwo, 2015) which means that they can be used to replace fossil fuels that have negative environmental impacts including greenhouse gas emissions. Before producing this material (FAME) in the plant, it is very necessary that a prototype plant of its production be setup and simulated using a process simulator like Aspen HYSYS in order to have an idea of how its production will be in real time.

According to the information obtained from the literature, some scientists and engineers have carried out some researches related to the subject matter of this work. For instance, Karacan and Karacan (2014) used aspen HYSYS for the simulation of reactive distillation used for the production of a fatty acid methyl ester at optimum conditions. According to the work, canola oil and methanol were used as feedstocks while potassium hydroxide and potassium methoxide were used as different formulations of catalysts. The three optimization algorithms (Fletcher-Reeves, Quasi-Newton and Successive Quadratic Programming (SQP)) investigated were found to produce relatively similar maximized mass fractions, which was the objective function of the optimization, of methyl oleate in the bottom section of the column. Simasatitkul *et al*. (2011) simulated reactive distillation for a fatty acid methyl ester production by transesterification of soybean oil and methanol using Aspen HYSYS, catalyzed by sodium hydroxide. The simulation results showed that a suitable configuration of the RD column consisted of only three reactive stages. Also, it was concluded from their work that methanol and soybean oil should be fed into the column in the first stage. The optimal operating conditions obtained in the work were molar feed ratio of methanol to oil of 4.5:1, molar reflux ratio of 3, and a reboiler duty of 1.6×10^7 kJ h^{-1}. Furthermore, Martins *et al*. (2013) worked on transesterification of soybean oil for oleic acid methyl ester production using hydrotalcite as basic catalyst. The reactions of the transesterification were carried out at atmospheric pressure and at a temperature of 64 °C in a jacketed reactor coupled to a condenser, under magnetic stirring, by varying the molar ratio (methanol/oil) and the reaction time. For the optimization of the simulation in HYSYS 3.2 process simulator, three different algorithms were used for the optimization; they are: Fletcher-Reeves, Quasi-Newton and Successive Quadratic Programming (SQP) algorithms. The objective function of the optimization was taken as maximizing the mole fraction of methyl oleate in the bottom stream of the column. In the work, it was concluded that Aspen HYSYS could be used to represent and simulate the process successfully. The three optimization algorithms investigated were found to produce relatively similar maximized mass fractions of methyl oleate in the bottom section of the column. Samakpong *et al*. (2012) simulated and optimized a fatty acid methyl ester production using reactive distillation of rubber seed oil, and they discovered that feedstock with high free fatty acids (FFAs) could not undergo transesterification with alkaline catalyst. However, the esterification of palmitic acid and methanol to biodiesel could be achieved via reactive distillation with 100% conversion and without feeding excess methanol. They also found that reactive distillation made hydrolysis (reverse) reaction to be negligible because water was constantly removed from the process. Giwa *et al.* (2014) investigated the performance of some fatty acids used for the production of fatty acid methyl ester in a reactive distillation column with the aid of Aspen HYSYS. The fatty acids considered were oleic acid, which was discovered, according to Kusmiyati and Sugiharto (2010), to give fatty acid methyl ester that had the quality required to be a diesel substitute, and some other ones (stearic acid, linoleic acid and palmitic acid) found to be present in jatropha oil. Methanol was used as the alcohol of the reaction. The results they obtained revealed that palmitic acid had the best performance in fatty acid methyl ester production.

It has been discovered that the work of Giwa *et al.* (2014) is worth considering for the production of fatty acid methyl ester. However, the optimization of the parameters required by the reactive distillation for operation has not been carried out.

Therefore, this work is aimed at determining the optimum parameters required for obtaining fatty acid methyl ester (FAME) of high purity in a reactive distillation column with the aid of Aspen HYSYS.

2. Methodology

The methods adopted in accomplishing this work, which was carried out to obtain the optimum parameters necessary for producing high purity fatty acid methyl ester and water, as the by-product, from the esterification reaction between palmitic acid and methanol via a reactive distillation process, are as outlined below.

2.1 Model Development and Simulation

To develop the model of the reactive distillation process for FAME production considered in this work, Aspen HYSYS V8.0 (Aspen, 2012) process simulator was employed. The chemical components (palmitic (a fatty) acid, methanol (an alcohol), methyl palmitate (an ester) and water) that were involved in the process were selected from the database of the simulator; Wilson model was chosen as the fluid package, and reaction set, which was taken to be an equilibrium type (Equation 1), was incorporated and added to the fluid package.

$$C_{16}H_{32}O_2 + CH_3OH \leftrightarrow C_{17}H_{34}O_2 + H_2O \quad (1)$$

After that, the reactive distillation column, which had two inlet streams and two outlet streams, was built as shown in Figure 1. The column comprised seven sections, viz, condenser section, rectifying section, fatty acid feed section, reaction section, alcohol feed section, stripping section and reboiler section.

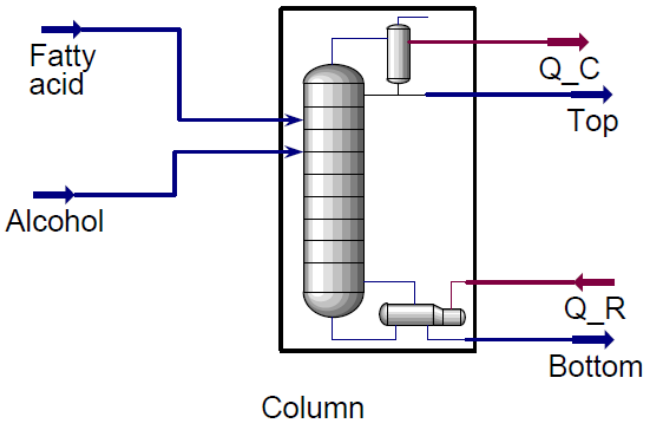

Figure 1: Aspen HYSYS model of reactive distillation process for FAME production

The other parameters used for the development and the simulation of the model of the reactive distillation process are given in Table 1.

Table 1: Model development and simulation parameters of the process

Parameters	*Values*
Palmitic acid feed temperature (^oC)	250
Palmitic acid feed pressure (atm)	5
Palmitic acid feed flowrate (kgmol/hr)	150
Methanol feed temperature (^oC)	150
Methanol feed pressure (atm)	1
Methanol feed flowrate (kgmol/hr)	165
Number of stages	17
Reflux ratio	3
Palmitic acid feed stage	6
Methanol feed stage	11
Reactive section	6-11
Condenser type	Total

Owing to the reactive distillation process involved, Sparse Continuation Solver was used as the algorithm for the simulation because it was the one that could handle this kind of a process.

2.2 Optimization

After the model of the reactive distillation process had been simulated, its optimization was carried out with the aid of the optimizer tool of the same Aspen HYSYS V8 used for the model development and simulation, which was accessed upon the addition of the "OptimizerSpreadsheet" unto the developed Aspen HYSYS model of the column as shown in Figure 2. The objective function of the optimization was taken to be the maximization of the mole fraction of the desired product (fatty acid methyl ester) in the bottom section of the column. Three optimizer algorithms were used, and they were Sequential Quadratic Programming (SQP), Box and Mixed algorithms.

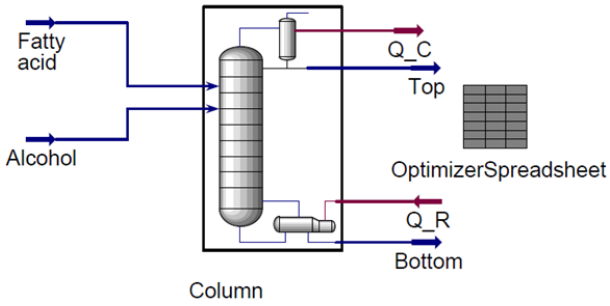

Figure 2: Aspen HYSYS reactive distillation optimized process flowsheet

The ranges of the manipulated variables used for the optimizations were as shown in Table 2.

Table 2: Ranges of the manipulated variables used for the optimization

Parameter	**Low bound**	**High bound**
Reflux ratio	1.5	6
Feed Ratio (kgmol/hr palmitic acid/kgmol/hr methanol)	0.5	2.2
Reboiler duty (kJ/hr)	9.00e+05	3.60e+06

At the end of the optimization, the obtained optimum values were used to simulate the process, again, for validation.

3. Results and Discussion

The results obtained from the simulation of the developed model of the reactive distillation process used for the production of the fatty acid methyl ester (methyl palmitate (desired product)) and water (by-product) from the esterification reaction between palmitic acid and methanol were as given in Figures 3 – 7. In the figures, stage numbers 0 and 18 refer to the condenser and the reboiler, respectively.

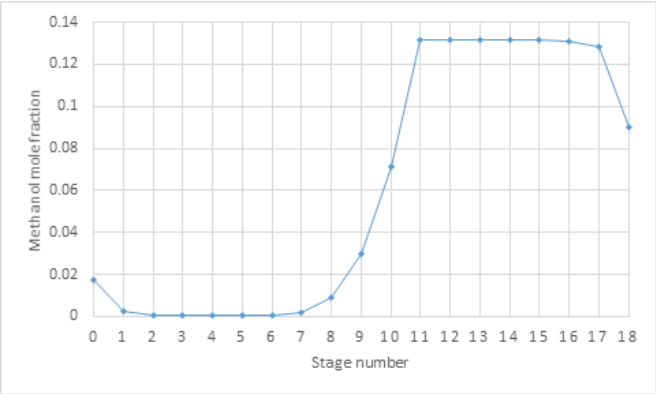

Figure 3: Mole fraction profile of methanol of the simulated process

Shown in Figure 3 is the variation of mole fraction of methanol with the stages of the column. According to the figure, methanol was found to have its highest mole fraction at the stripping section while its lowest mole fraction was at the rectifying section of the column. Also, the mole fraction of methanol was found to decrease upward after being fed at the 11th stage of the column. This was discovered to be as a result of the fact that methanol was being consumed as it was moving upwards the column within the reaction section.

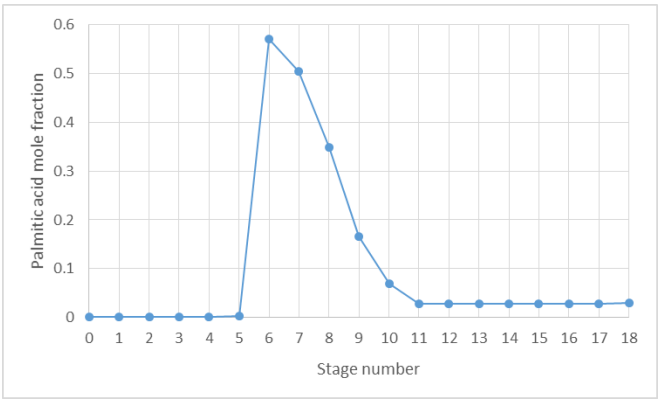

Figure 4: Mole fraction profile of palmitic acid of the simulated process

Figure 4 presents the mole fraction profile of palmitic acid. Palmitic acid was fed into the column at stage 6, and from there, towards the methanol feed section, a decline in its mole fraction was observed to take place. This was also found to be due to the reaction occurring between palmitic acid and methanol feed stages encompassing the reaction section of the column. Also observed from the figure was that the there was no palmitic acid in the top product as the unreacted palmitic acid were going down towards the bottom section of the column.

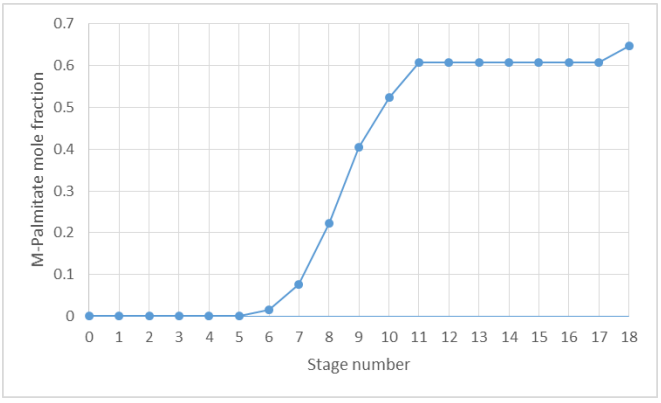

Figure 5: Mole fraction profile of methyl palmitate of the simulated process

Shown in Figure 5 is the mole fraction profile of the desired product (methyl palmitate) of the process. As can be seen from the figure, no methyl palmitate was found to exist at the condenser and the rectifying sections of the column because it was the least volatile of all the components involved in the process, as can be seen from their basic properties, especially the boiling points, given in Table A1 of the Appendix. Looking at the figure, the mole fraction of methyl palmitate was found to increase from the palmitic acid feed section towards methanol feed section. This was found to be an indication of methyl palmitate production at the reaction section due to the conversion of the reactants. It was also noticed that the mole fraction profile of the product (methyl palmitate) was constant at the stripping section. Also discovered from the results obtained was that the highest mole fraction of the product was in the reboiler section of the column, from where it (the product) was collected.

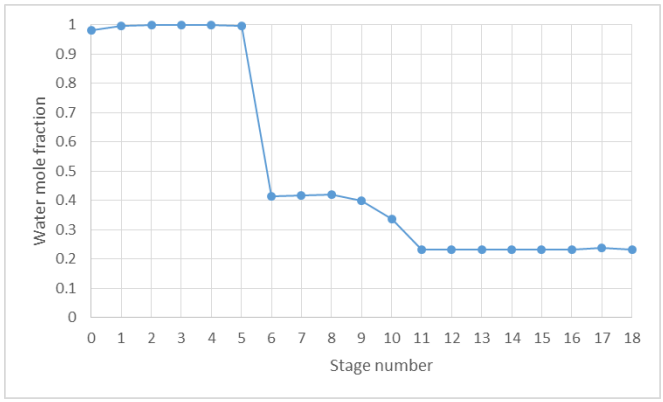

Figure 6: Mole fraction profile of water of the simulated process

Also estimated from the developed model of the process, and the mole fraction of which is shown in Figure 6 is the mole fraction of the by-product (water) of the process. From the figure, it was seen that water was the component that dominated the rectifying section of the column. From that (rectifying) section, towards the reaction section of the column, the mole fraction of water was found to decrease and, later, became constant at the stripping section of the column.

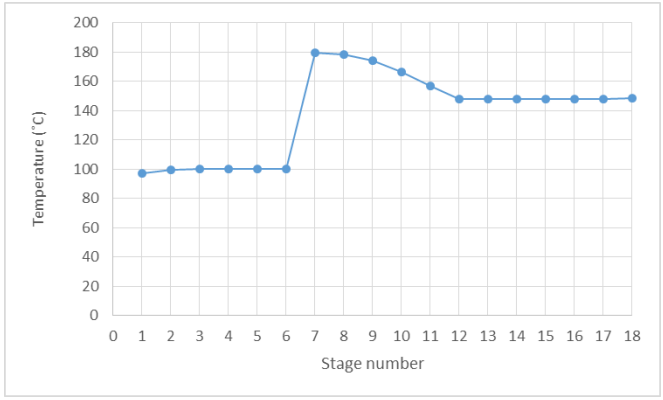

Figure 7: Temperature profile of the simulated process

Considering the temperature profile of the process, which is shown in Figure 7, it was found that the highest temperature of the process occurred at the reaction section. This was due to the exothermic nature of the reaction. As expected, the lowest and the highest temperatures of the process were observed to occur at the condenser section and the reboiler section, respectively.

It can be noticed that the highest mole fraction of methyl palmitate obtained from the simulation carried out was 0.648. The value was, actually, favourable. However, it was desired to have a product with higher purity. That, thus, necessitated carrying out the optimization of the process.

The results obtained from the optimization carried out using three different optimization algorithms (Box, Mixed and Sequential Quadratic Programming (SQP)) are given in Table 3. According to the table, taking the steady-state mole fraction value as the initial point, the optimization algorithm that gave the highest mole fraction of methyl palmitate of 0.7995 was found to be Box. In addition, SQP algorithm

yielded a very close value (0.7990) of methyl palmitate mole fraction to that of Box. The optimized value given by mixed algorithm was also found not to be too different beyond acceptable value from the other two. The differences in the values of the objective functions given by the three algorithms were accounted for by the differences in the optimum operating conditions given by each of them.

Table 3: Optimum parameter values

Parameter	Steady state	Box	SQP	Mixed
Reflux ratio	3	1.5	1.5	1.5
Reboiler duty (kJ/hr)	1.8e+06	2.626e+06	1.8e+06	1.8e+06
Feed ratio	1.1	1.181	1.124	1.297
Objective function	0.648	0.7995	0.7990	0.7893

Furthermore, the optimized values obtained from Box algorithm, which gave the highest value of the objective function (mole fraction of methyl palmitate) was used to simulate the process again for validation and the results obtained as the optimized profiles were compared with those of the simulated process, as shown in Figures 8 – 12.

From Figure 8, it was noticed that more methanol was present at the reaction section of the optimized process as compared to that of the initial simulation. This implied that the conversion with respect to methanol for the optimized process was less than that of the initial simulation. In other words, less methanol was being used up in the reaction section of the optimized process and, as such, more was leaving at the top section and slightly more was leaving from the bottom section of the column of the optimized process. This behavior of less methanol being used up in the reaction section was as a result of the slight increase in the feed ratio of the optimized process compared to that of the initial simulation.

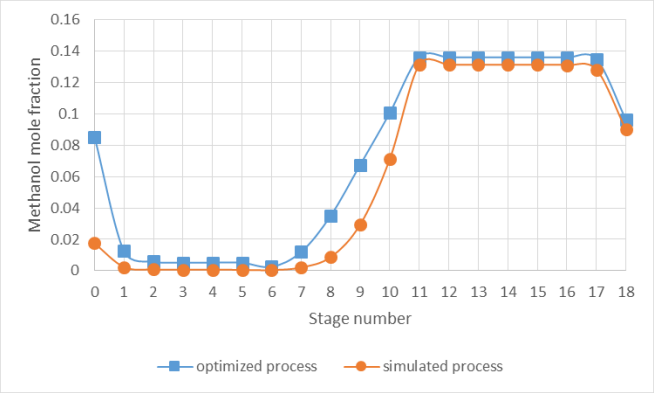

Figure 8: Comparison of simulation and the optimization mole fraction profiles of methanol

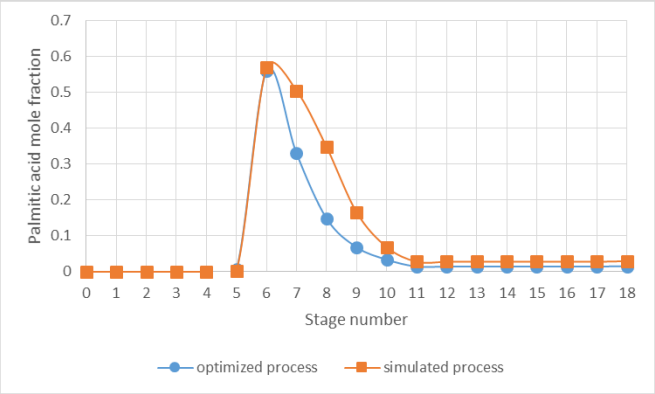

Figure 9: Comparison of the simulation and the optimization mole fraction profiles of palmitic acid

Figure 9 presents the variation of mole fraction of palmitic acid with stages of the column for the initial simulated and the optimized processes. From the figure, it was observed that the conversion of the optimized process with respect to palmitic acid was higher than that of the initial simulated one because in the optimized process, more palmitic acid was observed to disappear at the reaction section. It can also be seen that the mole fraction of palmitic acid at the bottom section of the column for the optimized process was less than that of the initial simulation due to high conversion of the optimized process, even though their profiles at the upper sections (condenser and rectifying) were approximately the same.

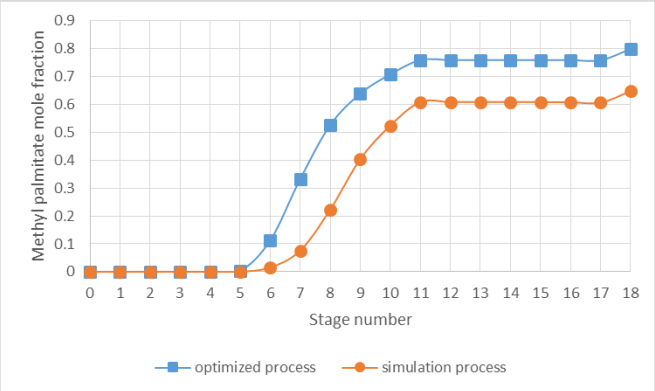

Figure 10: Comparison of the simulation and the optimization mole fraction profiles of methyl palmitate

From Figure 10 that is showing the profiles of the desired product (methyl palmitate), it was found that the mole fractions of methyl palmitate for the optimized process were higher than those of the initial simulated one in all the sections except at the rectifying and the condenser sections. Since the desired product was obtained from the bottom section of the column, it then means that the optimized process was able to give a higher product purity than that of the initial simulation, which was very desirable.

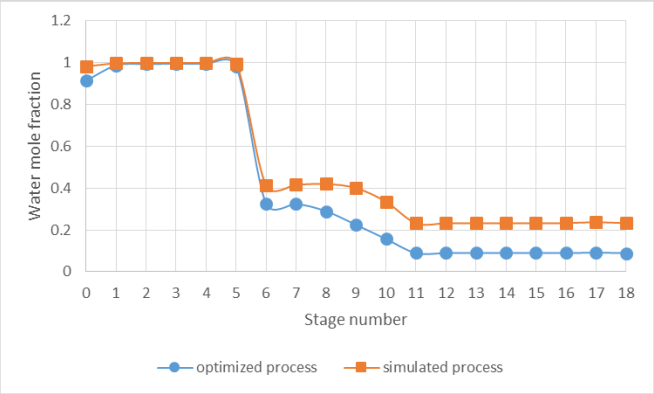

Figure 11: Comparison of the simulation and the optimization mole fraction profiles of water

The other profile (given in Figure 11) considered in this work was that of the water given as the by-product of the process. According to the figure, the mole fraction of water obtained from the initial simulated process was found to be higher than that of the optimized one. This was in indication that less water was being produced in the reaction section of the column of the optimized process than in the initial simulated one.

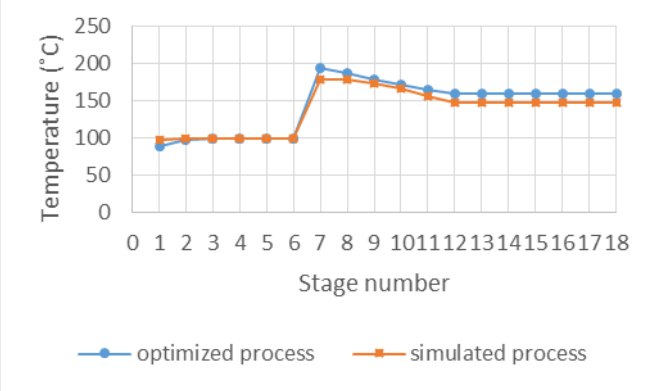

Figure 12: Comparison of the simulation and the optimization temperature profiles of the process

Shown in Figure 12 is the comparison between the temperature profiles of the optimized and the simulated process. From the figure, it was found that the highest temperatures of both of the processes occurred at the reaction section of the column, as discussed earlier in the case of the simulated process. Also noticed was that the temperatures of the optimized process (using Box algorithm), especially from the reaction section down to the reboiler, were higher than those of the simulated one. This could be seen from the higher reboiler duty of the optimized process than that of the simulated one.

4. Conclusion

The results obtained from the simulation of the reactive distillation process used to produce methyl palmitate (a fatty acid methyl ester (FAME)) carried out in this work revealed that FAME can be produced successfully using reactive distillation process because the developed model was able to converge when simulated to give a FAME product having a mole fraction of 0.648. Furthermore, the optimization of the process revealed that higher purity than that (0.648) could be obtained using Box optimization algorithm because the result of the objective function, which was the maximization of the mole fraction of the FAME obtained from the bottom section of the column was achieved to be 0.7995. The results given by the simulation of the process using the optimum values (reflux ratio of 1.5, feed ratio of 1.181, and reboiler duty of 2.626e+06 kJ/hr) obtained showed that they were theoretically valid. Finally, it has been seen that the Aspen HYSYS model developed in this research work can be used to represent, simulate and optimize a reactive distillation process successfully. It is recommended that, at least, an experiment should be carried out to experimentally validate the optimum values of the manipulated parameters obtained in this work.

5. Acknowledgement

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6. Nomenclature

Bottom Bottom product
FAME Fatty acid methyl ester
M-palmitate Methyl palmitate
Q_C Condenser duty (kJ/hr)
Q_R Reboiler duty (kJ/hr)
RD Reactive Distillation
SQP Sequential Quadratic Programming
Top Top product

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Appendix

Table A1: Some basic properties of the process components

Component	Molecular weight (kg/kgmol)	Boiling point (°C)	Density (kg/m^3)
Palmitic acid	256.4	351.0	881.6
Methanol	32.04	64.65	795.7
Methyl palmitate	270.5	326.1	880.0
Water	18.02	100.0	998.0

Source: Aspen, 2012

Author Profile

Samson Chukwufumnanya NWAMBUONWO was born in Lagos State, Nigeria in 1992. He received his Bachelor Degree in Chemical Engineering from Afe Babalola University, Ado-Ekiti, Ekiti State, Nigeria in 2015 with *First Class Honours*. He had his internship programme of his undergraduate programme under Pan Ocean Oil Cooperation University Internship Programme (Scholarship) where he learnt about gas and crude oil processing in the Pan Ocean gas plant processing facility and flow station.

Dr. Abdulwahab GIWA was born in Ile-Ife, Osun State, Nigeria in 1976. He obtained his National Diploma in Chemical Engineering from Kaduna Polytechnic, Kaduna, Nigeria in 1998 with a *Distinction* Grade as the Best Student of the Programme. Furthermore, he received his Bachelor Degree in Chemical Engineering from Federal University of Technology, Minna, Nigeria in 2004 with *First Class Honours* as the Best Student in School of Engineering and Engineering Technology. Moreover, he got his PhD Degree from Ankara University, Ankara, Turkey in 2012, also in the field of Chemical Engineering, with a Cumulative Grade Point Average (CGPA) of *4.00 out of 4.00*. Thereafter, he proceeded to Middle East Technical University, Ankara, Turkey to have his Postdoctoral research experience. He is currently a Senior Lecturer with the Department of Chemical and Petroleum Engineering, Afe Babalola University, Ado-Ekiti, Ekiti State, Nigeria. He is very interested in researches in the areas of Process Modelling, Simulation, Optimization, Design and Control.