Dealcoholization of Palm Wine Using Osmotic Membrane Distillation

Ejikeme Ebere M., Prof. Igbokwe P.K., and Ejikeme P.C.N
Met Department Proda, Enugu, Nigeria
Chemical Engineering Department, Nau Awka, Nigeria
Chemical Enginnering Department, Esut Enugu, Nigeria

Abstract– **In this study, the possibility of using osmotic membrane distillation in reducing the alcohol content of palm wine was investigated. The effect of various process parameters such as temperature (30–40^{o}C), stirring speed (0 – 100rpm) and membrane pore size (0.2–0.45μm) was studied. All the experiments were carried out on a membrane cell mode using hydrophobic polytetrafluroethylene membrane (PTFE). The design of experiments and its analysis was done using a design expert 8.0.2.Software. The statistical analysis done showed that the three factors studied had significant effects on the flux which is rise in water level. Flux was directly proportional to the temperature and stirring speed effects and inversely proportional to the membrane pore size effect. It was observed that of all the factors investigated, stirring speed had the highest effect with 29.3% increase followed by temperature with 20.4% increase and the least parameter was membrane pore size with 8.6% decrease on flux. It was deduced that flux which is the rise in water level is directly proportional to the amount of alcohol removed. The model equation was also obtained while the model adequacy check done revealed that the equation can adequately explain the process**

Keywords– Design of Experiment, Flux, Osmotic Membrane Distillation and Palm Wine

I. INTRODUCTION

Palm wine is an alcoholic beverage obtained from the sap of Raphia Vinifera, Raphia Hooderi (raphia palm) and Elaeis guineensis (oil palm) [1], [2], [3]. The alcoholic content of palm wine varies depending on the degree of its fermentation. The fresh sap contains low proportion of alcohol (only about 2.3%) and ferments quickly by the action of bacteria and natural yeast to produce a more piquant drink (milky flocculent appearance with a slightly sulphurous odour) [4].

Palm sap begins fermenting immediately after collection, due to natural yeasts in the air (often spurred by residual yeast left in the collecting container). Within 2 hours, fermentation yields an aromatic wine of up to 4% alcohol content, mildly intoxicating and sweet [5]. The wine may be allowed to ferment for a longer time up to a day, to yield a stronger, more sour and acidic taste which some people prefer [5].

Since palm wine has a high alcoholic content, excessive consumption of it has both health and socio-economic implications. Therefore, a small adjustment in the alcohol content is currently and recently one of the most important objectives. A method of removing some of the alcohol content will allow the wine to ferment longer with the optimum flavor and fragrance maturity which some people prefer without suffering the negative effect of excessive alcohol.

There are several methods disclosed in the art for reducing the alcohol content of fermented beverages, however, each process has its advantages and disadvantages in terms of process cost and product quality.

The simplest method is arresting the fermentation earlier; this will lead to production of low alcoholic wine but will not give that stronger, sour and acidic taste which some people prefer. Therefore, a method is needed which will reduce the alcohol content still retaining the flavor and fragrance of the wine.

Membrane processes can be utilized for the dealcoholisation process. Membrane processes mainly belong to the group of processes with no heat impact on the product. They are either pressure-driven (i.e., reverse osmosis) or concentration – driven (i.e., dialysis processes) [6]. In this work, osmotic membrane distillation, which is driven by partial pressure gradient over the membrane, was used. Osmotic membrane distillation is a novel process that uses membrane to remove the alcohol content of fermented beverages still retaining the flavor and fragrance components of the wine [7]. OMD uses a hydrophobic micro porous membrane, which separates the two aqueous solutions, one being the feed or dilute solution and the other being the osmotic agent (OA) or brine solution of different osmotic pressure.

II. ADVANTAGES OF OMD

OMD offers major advantages in comparison with other processes [7].

This process is highly selective for the removal of alcohol relative to water because the vapor pressure of water over most alcoholic ferment is very nearly that over pure water.

The lower temperature employed can help avoid chemical reactions associated with heat treatment and prevent degradation of flavor, color and loss of volatile aroma.

Only volatile compound which can permeate the membrane will be separated and non volatile solutes such as ions, sugars, macro molecules, cells and colloids are totally retained in the feed.

The transport rate of flavor/fragrance components from wine to strip solution is reduced because the solubility of these components in alcohol/water solution are substantially higher (and their vapor pressure lower) than they are in plain water.

The aim of this work is to study the viability of using OMD in dealcoholisation of palm wine, to study the effect of process factors like temperature, stirring speed and membrane pore size on the dealcoholisation process and also to fix a model that will adequately explain the process.

III. METHODOLOGY

A. Palm wine

A freshly tapped palm wine was bought from a local Emene market in Enugu State Nigeria. It was allowed to ferment for some hours.

B. Membrane

A circular hydrophobic polytetrafluroethylene (PTFE) membrane of 0.45µm and 0.25µm porosities and 142mm in diameter was bought from Sartorius Stedium Biotech Germany.

C. Distilled Water

Extra pure distilled water was bought from pyrogen free Water Company Setdeo Nigeria Limited New Haven Enugu, Enugu State, Nigeria.

D. Sample Preparation

The palm wine sample was stabilized by arresting the fermentation using a combined preservation method. This method involved pasteurization followed by chemical treatment. The sample was analyzed for alcohol 24hrs and 48hours after stabilization to ascertain the efficacy of the process.

E. Determination of Alcohol Content

The alcohol determination was done using distillation method, followed by specific gravity determination as in [8].

F. Flux Determination

This was done using the method described in [10]. The flux determination apparatus consisted of two glass reservoirs of equal volume (2L) labeled A and B. A was the feed reservoir with side arm at the base and was connected to osmotic membrane cell B with side arm at the base through a Teflon tube. A known quantity of beer was introduced into ‘A through the open vent and the beer flowed freely by hydrostatic pressure into B until the sample in reservoir B touched the membrane unit.

Magnetic bar was introduced into reservoir B and the reservoir was mounted on the magnetic stirrer hot plate. Reservoir A was equally mounted on a hot plate so that the two reservoirs were on the same level to cancel the effect of hydrostatic pressure changes due to difference in levels. The two reservoirs were placed on the same temperature.

In reservoir B, distilled water was introduced through the upper vent so that there were two fluid compartments (water and wine) separated by the membrane unit and the vent in B closed tightly to arrest vaporization.

The rise in height of water in cell side was measured every 1hr for 4 hours with the aid of a meter rule attached to the upper part of reservoir B. The corresponding flux was calculated.

The system was subjected to different variables according to the design layout in standard order generated by the Design – expert software in table 2 and their corresponding fluxes were calculated. Table 1 shows the factors and levels of two factorial design employed in the experiment.

IV. RESULTS AND DISCUSSION

The results obtained according to the full factorial design layout generated by Design Expert was analysed to obtain the model equation.

The effects that were included in the model were selected using half normality plot in Fig. 1.

The half normality plot showed that the factors of temperature, stirring speed and membrane pore had significant effects on flux.

Also the Pareto Chart (Fig. 2) which displayed the magnitude of each effect showed that stirring speed had the highest effect, followed by temperature and lastly membrane pore size.

The analysis of variance (ANOVA) (table 3) showed that the model terms are significant.

The F-value of 95.58 implied that the model is significant, values of prob > F less than 0.0500 indicated that the model term is significant. In this case A, B, and C are significant model terms. Values greater than 0.1000 indicated that the model terms were not significant.

The model equation in terms of coded value:

$$\text{Flux} = +1.03 + 0.12*A + 0.18*B - 0.15*C \quad \ldots\ldots\ldots\ldots\ldots \quad (1)$$

Final model Equation in terms of Actual factors:

$$\text{Flux} = +0.39365 + 0.024325*\ \text{Temperature} + 3.50250E-003*\ \text{Stirring Speed} - 1.18700*\ \text{pore size} \quad \ldots\ldots\ldots\ldots \quad (2)$$

A. Model Adequacy Check

The examination of fitted model was done to determine whether it adequately gave the approximation of the response surface. The model diagnostic plots were used which mainly displayed residuals.

Some of the diagnostic plots used are: The normal probability plots which followed a straight line showing that the residuals followed normal distribution.

Plots of residuals vs. the ascending predicted response values showed a random scatter which indicted that there was constant range of residual across the graph.

The plots of residuals vs. experimental run order viewed a random scatter which indicted that there were no lurking variables that can affect the process.

The graph of predicted response values vs. actual response values was split evenly by the 45 degree line which showed that there was no group of value(s) that was not predicted by the model. The analysis showed that there was no deviation from assumptions made by ANOVA. Therefore, the model selected was adequate.

B. Temperature Effect

The plot of temperature effect on flux (Fig. 3) showed that it had positive effect on flux.

The increase of temperature from 30^0C to 40^0C had 20.4 percent increase. It is known that mass transfer in many transport processes shows Arrhenius dependency on temperature [9]. Similar behavior was observed in the present study due to the fact that the activity coefficient of the strip solution remained constant over the range of temperatures studied. Small rise in temperature provided extra driving force which in turn increased the activity coefficient of the feed solution.

C. Stirring Speed Effect

The experiment was done with zero stirring and equally with stirring at 100rpm. The effect plot (Fig. 4) showed that it had positive effect on flux.

It had 29.3 percent increase on flux. This was possible because stirring provided mild agitation of the feed solution thereby reducing the effect of membrane fouling and concentration polarization [10]. Membrane fouling results from the irreversible blocking of membranes by adhesion of insoluble compounds to the membrane matrix reducing the rate of flux.

D. Membrane Pore Size Effect

This experiment was done with 142mm diameter flat circular hydrophobic polytetrafluroethylene membrane of 0.2µm and 0.45µm porosities. The single effects plot (Fig. 5) showed that it had negative effect on flux. It showed that membrane at its lower level resulted to higher mean response compared to that at its higher level.

The increase in membrane pore size resulted to decrease in flux by 22.8%. This was in agreement with the literature that small diameter fiber membrane with thin walls appears to be the best candidate for osmotic membrane distillation because they offer higher area to volume ratio and has a higher pore entry pressure.

E. Three Factors Interaction Effect

To obtain the interaction effects of the three factors: temperature, stirring speed and membrane pore size, cubic plot was used. It showed the combinations of the levels of the factors that can give both minimal and maximal fluxes.

It showed that the flux was maximum at setting C-, B+,A+ at the upper front right corner with value of 1.480 and minimum at setting B-, A-, C+ at the lower back left corner with value of 0.589.

Therefore, the experiment done at the maximum settings will give optimal results.

V. CONCLUSION

The entire analysis led to the conclusion that dealcoholisation of palm wine can be done successfully with osmotic membrane distillation process.

Stirring speed had highest effect on flux with 29.3% increase, followed by temperature with 20.4% increase and lastly membrane pore size with 8.34% increase.

It was therefore concluded that flux is directly proportional to temperature and stirring speed and inversely proportional to the membrane pore size.

Since it was obtained that flux which is the rise in water level increased as the alcohol removal was increased, it was therefore concluded that flux is directly proportional to the amount of alcohol removed.

VI. RECOMMENDATIONS

Dealcoholisation using OMD also removes to an extent some other volatile components of wine.

It is recommended that the strip solution be spiked with these compounds so that no concentration gradient for the compound exists.

Ethanol removed should be recovered from the strip solution by rectification. This can serve as a potential blending stock for production of fortified wines, liqueurs and whiskeys.

REFERENCES

[1] Bassir O. Some Nigerian Wines. West Afr J Biol Appl Chem 1968; 10, pp: 41–45.

[2] Faparusi SI. A biochemical study of palmwine from different varieties of Elaeis quineensis. PhD Thesis 1966, University of Ibadan, Nigeria.

[3] Okafor N. The Source of the micro-organisms in palmwine Sym Nig Soc Microbiology 1972; 1:97

[4] Tercula Mkpele et al (2011). The effect of palmwine on the morphometry and femur Histology of Westar Rat Foetuses. Journal of Medicine in the Tropics (2011) 13:@:129 – 133.

[5] Palmwine – from Wikipedia, the free encyclopedia.

[6] Erlangung des Grades (2008) prozessbewertungder osmotischen Destillation Zur Entalkoholisierung Von Wein – PhD Thesis.

[7] P.A. Hogan, R.P. Canning, P.A. Peterson, R.A. Johnson and A.S. Michaels, A new option: osmotic distillation, Chem. Eng. Prog., 94 (1998), pp: 49–61.

[8] Brewing Process: Packed beer-Alcohol Distillation method (EBC Method).

[9] J. M Coulson, and J. F Richardson, ‘Fluid flow, heat transfer and mass transfer in Chemical Engineering’ (2004), Vol. 1, 6th Edition.

[10] A.V. Narayan et al. ‘Acoustic field – assisted Osmotic membrane distillation’147 (2002), pp: 149 –156.

Table 1: Factors and levels of full factorial design

FACTORS	**UNITS**	**LOW LEVEL (-)**	**HIGH LEVEL (+)**
A: Temperature	^{O}C	30	40
B: Stirring speed	Rpm	0	100
C: Membrane size	Nm	0.2	0.45

Table 2: Design layout in standard order response data entered

Standard Order	**Run Order**	**Factor 1 Temp (^{O}C)**	**Factor 2 Stir. Speed (rpm)**	**Factor 3 pore size(µm)**	**Flux L/m^{2}.hr**	**Final concentration (%m/m)**
4	1	40	100	0.2	1.531	7.11
8	2	40	100	0.45	1.182	7.370
3	3	30	100	0.2	1.218	7.290
2	4	40	0.000	0.2	1.083	7.450
6	5	40	0.000	0.45	0.829	8.320
5	6	30	0.000	0.45	0.625	8.500
1	7	30	0.000	0.2	0.900	8.100
7	8	30	100	0.45	0.909	7.88

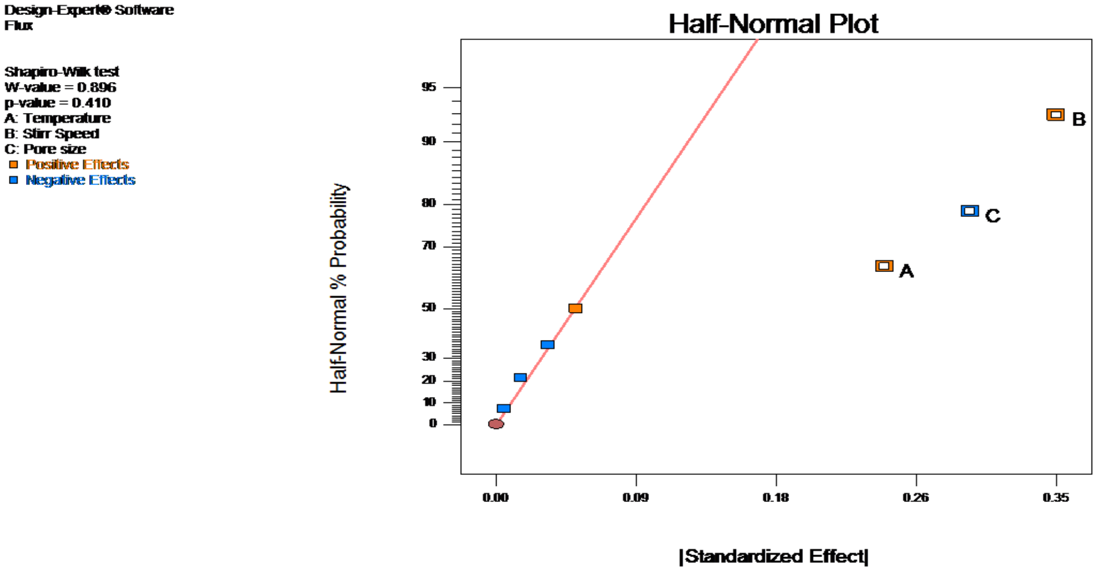

Fig. 1: Half normal plot – all big effects selected

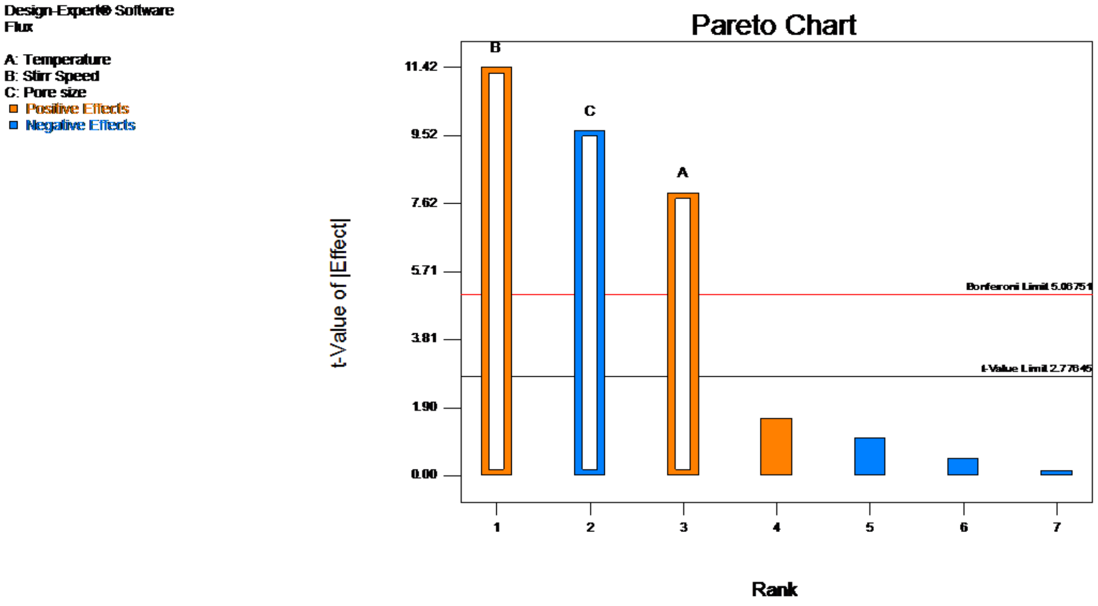

Fig. 2: Paretto Chart of Effects

Table 3: ANOVA Table

Response 1 Flux

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob > F
Model	0.54	3	0.18	95.58	0.0004 Significant
A – Temperature	0.12	1	0.12	62.78	0.0014
B – Stirring Speed	0.25	1	0.25	1130.52	0.0003
C-Pore Size	0.18	1	0.18	93.43	0.0006
Residual	7.541E – 003	4	1.885E – 003		
Cor total	0.55	7			

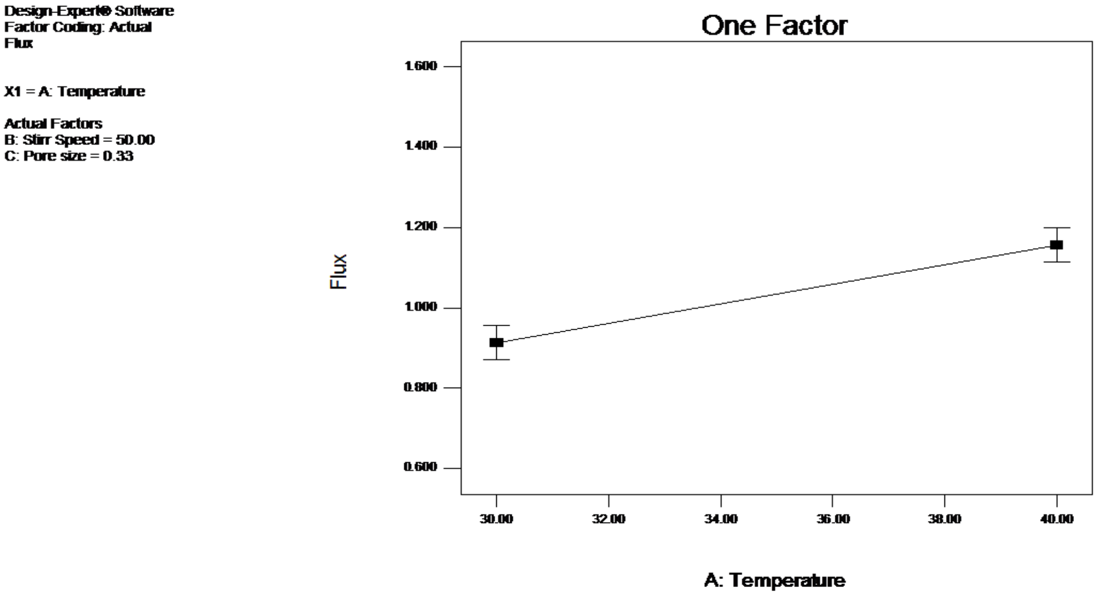

Fig. 3: Single effect of temperature

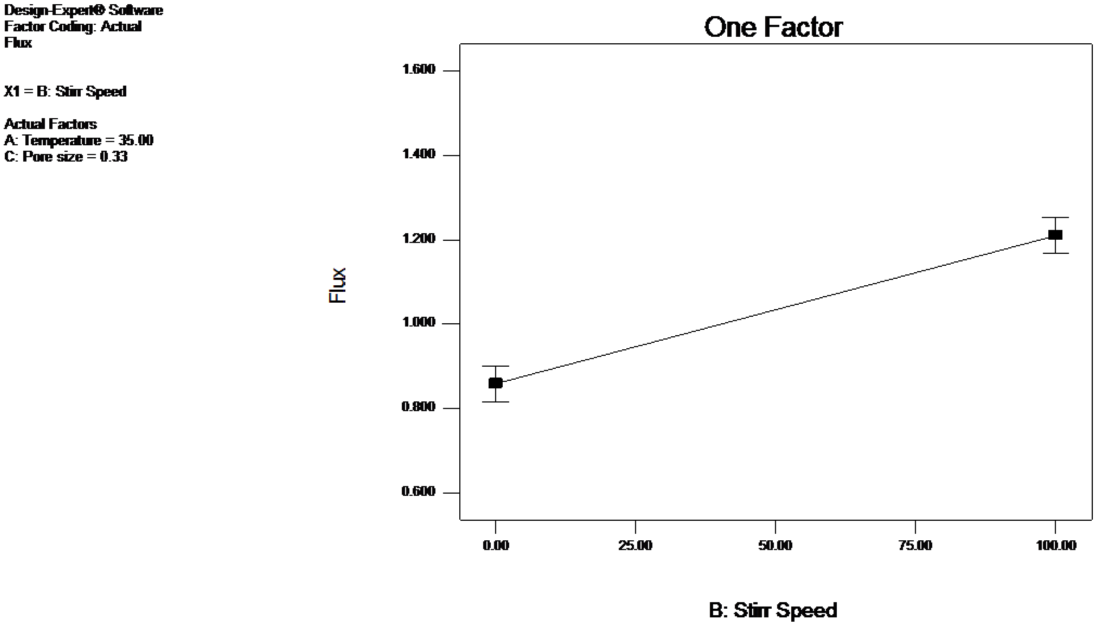

Figure 4 – Single effect of stirring speed on flux

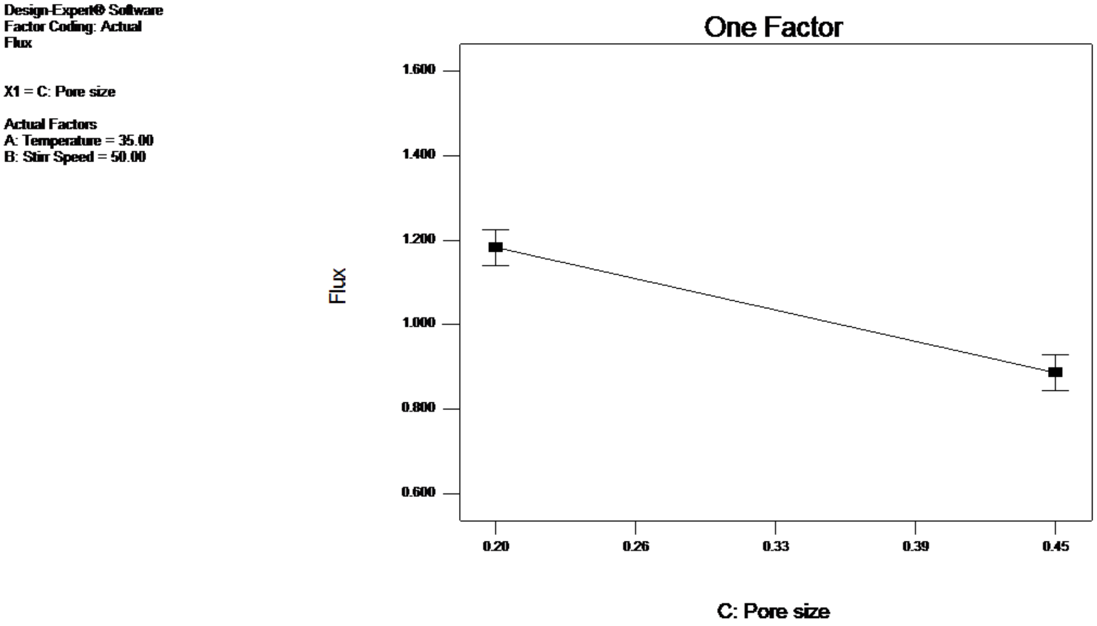

Fig. 5: Single effect of membrane pore size on flux

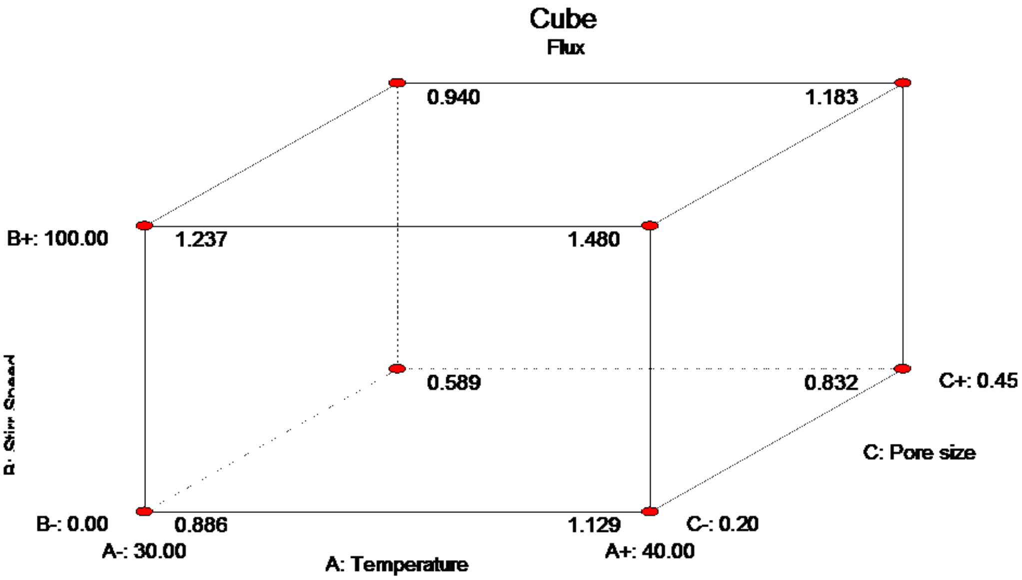

Fig. 6: Cubic Plot of the three factors