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Quadratic Model For Reduction of Alcohol Content of Palm Wine

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ABSTRACT

The aim of this work is to reduce the alcohol content of palm wine using osmotic membrane distillation process. Osmotic membrane distillation is a novel process that uses membrane to remove the alcohol content of fermented beverages without compromising the flavour and fragrance components of the wine. Hydrophobic polytetrafluoroethylene (PTFE) membrane was used for the distillation process. The palm wine was fermented for two days before stabilization. The stabilization method used was pasteurisation followed by chemical treatment with Sorbic acid. It was observed that the alcohol content increased from 8.2% to 25% after two days of fermentation and remained constant after two days of stabilization. This confirmed the efficacy of the stabilization method used. Sugar content was found to decrease from 6%brix to 4.9%brix after fermentation. Central composite design (CCD) was used for the optimization of three process factors; time, temperature, and stirring speed while reduction in percentage alcohol was used as the response. It was observed that the single effects, interaction effects and quadratic effects of time, temperature and stirring speed were significant. Quadratic model was developed and diagnosed for the alcohol reduction process. The optimum conditions generated were time of 3hrs, temperature of 45^oC, and stirring speed of 150rpm with predicted alcohol reduction of 55.2%. The validated conditions gave experimental alcohol reduction of 55.3% with 0.18% deviation from the predicted value.

Keyword: *CCD, Palm wine, Pasteurisation, PTFE, fermentation,*

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I. INTRODUCTION

Palm wine also known as palm toddy or “toddy” is a fermented drink made from the sap collected from virtually any species of palm tree. It is collected (or tapped) from tall trees, or raffia tree which is shorter and thus more accessible. There are reports that some people also collect sap from oil palm trees (Elaies guineensis). However, sap from a palm tree (raffia tree) is milkier and sweeter. Fermentation begins soon after the sap begins flowing. Within 1 to 2hours the alcohol content may reach up to 4%. Continued fermentation for up to 24hours results in a more alcoholic, acidic, highly flavoured and sour white drink.

Aroma and flavour are undoubtedly critical to the appeal of wine to the consumer, together with color, taste and mouth feel [1]. The human preference for aroma and flavour in palm wine is generally self evident. Such aroma and flavour come from well fermented wine which undoubtedly results to high alcohol content. Alcohol concentration of wine is important for various reasons. Besides its psychological and physiological effect on health, ethanol is indispensible for the aging, stability and organoleptic properties of wine [2, 3, 4], but it can also influence our perceptions of astringency (due to tannin), sourness (due to acid), sweetness, aroma and flavour [5, 6]. additionally, wine with high alcohol content are taxed at a higher rate in many countries [7] these reasons in concert with current health concerns related to elevated alcohol consumption [7], and growing market demand have led the wine industry actively seeking ways to facilitate the production of wine with lower alcohol concentration [7] without compromising wine flavour, consumer acceptance or increasing cost of production.

Wines of reduced alcohol content have been classified as dealcoholized or alcohol free (< 0.5 % v/v), low alcohol (0.5 to 1.2% v/v), reduced alcohol (1.2 to 5.5–6.5 % v/v) and lower alcohol wine (5.5 to 10.5 % v/v) [8, 9].

There are many methods that can be used to reduce the alcohol content of wine. The simplest method is arresting the fermentation on time. This leads to wine with lower alcohol content, however, it can compromise the full bodied character, aroma and flavour that are desirable in many wine which manifest on full fermentation. Another approach is to add water to the wine. While this has been practiced for centuries, it diminishes wine quality by reducing the overall concentration of wine. It is also illegal in many jurisdictions [10]. Other methods are modification of fermentation process [11], vacuum distillation, spinning cone column, freeze concentration [12], pervaporation [13], reverse osmosis [14], and osmotic distillation [14].The membrane process can also be utilized for the reduction of alcohol content and it allows the alcohol content to be reduced under mild condition so that organoleptic feature will remain unchanged.

In this work, osmotic membrane distillation process was used to reduce the alcohol content of palm wine using polytetrafloroethylene membrane. Osmotic membrane distillation uses a hydrophobic micro porous membrane which separates the two aqueous solutions one being the feed and the other being the osmotic agent of different osmotic pressure [15].

II. MATERIALS AND METHODS

2.1 Materils

Newly tapped palm wine was ordered from a tapper at Emene market Enugu, Enugu State Nigeria. Circular hydrophobic polytetrafloroethylene membrane was bought from Sartorous Stedium Biotech Germany. Distilled water was purchased from Pymotech research center Abakpa, Enugu State Nigeria.

2.2 Stabilization Of Palmwine

The fresh palm wine was analysed for specific gravity, pH, and alcohol content immediately after collection and was allowed to ferment for 48hour. The rate of fermentation was monitored on daily basis in term of the specified parameters. After 48hours, it was stabilised by arresting the fermentation using combined preservation method. This method involved pasteurization at 70^{o}C for 30mins, followed by chemical treatment using Sorbic acid. The efficacy of the preservation method was ascertained by determination of the alcohol content 24 and 48hours after stabilization.

2.3 Determination Of Alcohol Content Of Palmwine

The alcohol content of the palm wine was determined using distillation method. This involved distilling a known quantity of the palm wine after which the alcohol content of the distillate was determined using alcohol meter

2.4. Dealcoholisation Process

The method used for the dealcoholisation process was according to the work done by Ejikeme et al. 2013 [16] with slight modifications. It was done using Osmotic membrane distillation cell and the reservoir. The flux determination apparatus consisted of two glass reservoirs of equal volume (2litre) labelled 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 stabilized palm wine was introduced into A through the open vent and palm wine flowed by hydrostatic pressure into B until the alcohol in reservoir B touched the membrane unit. Magnetic bar/follower was introduced into reservoir B and the reservoir B was mounted on the hot plate magnetic stirrer. Reservoir A was equally placed 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 (3ml) 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 with water bound to arrest vaporization.

The hotplate magnetic stirrer was powered at 240V and stirring speed adjusted to desired speed. The rise in height of water in cell side was measured every 1 hr with the aid of a meter rule attached to upper part of reservoir B using the formula h_2-h_1 where h_2 represented the new water column and h_1 the original water column. The dealcoholisation process was strictly based on the design matrix on table 2. After each time interval, the alcohol content remaining on the feed was analysed using procedure specified on section 2.3 from which percentage alcohol reduction was calculated using the initial alcohol present. The values obtained were analyzed using Design-Expert 8.0.7.1 software to obtain the model equation.

2.5. Response surface method (RSM) for the optimization process

RSM is a collection of mathematical and statistical techniques useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and objective is to optimize this response [17]. Central composite design (CCD) is a type of RSM and is characterized by three operations; 2n axial runs, 2^n factorial runs and six centre runs. The total number of experiments is $2n + 2^n + nc$ where n is the number of factors and nc the number of centre points [18]. Three factors were optimized namely; time (hrs), temperature (°C), and stirring speed (rpm) and the response of interest which was optimized is the percentage alcohol removal. Factors and levels used for the optimization are shown on table 1, while the design matrix with the response is shown on table 2.

Table 1. Factors and levels used for optimization

Factors	Units	Levels				
		-α	-1	0	+1	+α
Time	Hrs	2.5	3.0	3.5	4.0	4.5
Temperature	°C	30	35	40	45	50
Stirring speed	Rpm	50	100	150	200	250

Table 2. Design matrix with the responses

Std Order	Run	Time	Temperature	Stirring speed	Alcohol removed
6	1	4.00	35.00	200.00	29.468
8	2	4.00	45.00	200.00	22.56
17	3	3.50	40.00	150.00	41.612
4	4	4.00	45.00	100.00	48.652
15	5	3.50	40.00	150.00	36.88
2	6	4.00	35.00	100.00	64.844
5	7	3.00	35.00	200.00	23.944
13	8	3.50	40.00	50.00	86.588
3	9	3.00	45.00	100.00	66.284
9	10	2.50	40.00	150.00	44.184
7	11	3.00	45.00	200.00	55.088
14	12	3.50	40.00	250.00	39.328
20	13	3.50	40.00	150.00	43.992
19	14	3.50	40.00	150.00	40.864
1	15	3.00	35.00	100.00	42.756
10	16	4.50	40.00	150.00	26.568
16	17	3.50	40.00	150.00	39.532
11	18	3.50	30.00	150.00	25.28
18	19	3.50	40.00	150.00	40.296
12	20	3.50	50.00	150.00	42.172

III. RESULTS AND DISCUSSIONS

3.1 Fermentation Of Palm Wine

The freshly collected palm wine was allowed to ferment for two days before arresting the fermentation. Immediately after collection, the sample was analysed for alcohol content (%), specific gravity and sugar content (%brix). To ascertain the efficacy of the stabilization method used, the palm wine was equally analysed on the first and second day after stabilization. Table 3 shows the result of the analysis of the fresh, fermented and pasteurised wine. It can be seen from the result that the alcohol content of the palm wine increased from 8.2% after collection to 25% after two days of fermentation. The same value was obtained on the first and second day after stabilization. This showed that pasteurisation and chemical treatment was effective in arresting the fermentation of the palm wine. It was equally observed that the sugar content decreased from 6% brix to 4.9 % brix after fermentation and remained constant two days after stabilization. The decrease could be attributed to the consumption of the sugar by the yeast with subsequent conversion to ethanol.

Table 3. Properties of palm wine during fermentation and after stabilization

Parameters	Fresh palm wine	1st day of fermentation	2nd day of fermentation	1st day after stabilization	2nd day after stabilization
Specific gravity	0.999	0.994	0.990	0.990	0.990
Alcohol content (%)	8.2	18.0	25.0	25.0	25.0
Sugar content (%brix)	6.0	5.3	4.9	4.9	4.9

3.2. Reduction Of Alcohol Content

Using the experimental set up for the dealcoholisation process, it was observed that the quantity of water on the upper layer of the first chamber directly in contact with the palm wine increased after each run. This increase in water layer can be attributed to the alcohol removed since the membrane is hydrophobic. Equally, it is highly selective for the removal of alcohol relative to water because the vapour pressure of water over most alcoholic ferment is very nearly that over pure water [15]. The removal of flavour and aroma components was assumed negligible because their solubility in the palm wine is substantially higher (and their vapour pressure lower) than they are in plain water. Other components of the wine which are not volatile cannot permeate the membrane and consequently retained in the feed. Table 2 shows the amount of alcohol retained after each run. The data was analysed using design expert software 8.0.7.1.

3.3. Selection Of Predictive Model For The Alcohol Reduction Process

Sequential model sum of squares was used to select the best model for response prediction based on the highest order model that was significant (small p-value) and not aliased, no lack of fit (p-value> 0.1) and reasonable agreement between adjusted R- squared and predicted R- squared (within 0.2 of each other). The summary of sequential sum of square for all the models is shown on table 4. From the table 4, quadratic model was selected based on the conditions stipulated.

Table 4. Summary table of sequential model sum of squares for alcohol reduction process

Source	Sequential p- value	Lack of fit p- value	Adjusted R-squared	Predicted R- squared
Linear	0.0038	0.0006	0.4754	0.1747
2FI	0.0510	0.0012	0.6375	0.4907
Quadratic	<0.0001	0.7158	0.9824	0.9638
Cubic	0.7248	0.3928	0.9782	0.7817

3.4. Analysis Of Variance (ANOVA) For Alcohol Reduction Process

Analysis of variance table (5) was used to test the selected model and the factors for significances based on the p- values. The Model F-value of 118.87 implied the model was significant. There was only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicated model terms were significant. In this case single effects of time (A), temperature (B), stirring speed (C), two factors interaction effects of time and temperature (AB), time and stirring speed (AC), temperature and stirring speed (BC), and quadratic effects of time (A^2), temperature (B^2), stirring speed (C^2) were significant model terms. Values greater than 0.1000 indicated the model terms were not significant. The "Lack of Fit F-value" of 0.58 implied the Lack of Fit was not significant relative to the pure error. There was a 71.58% chance that a "Lack of Fit F-value" this large could occur due to noise.

Table 5. Analysis of variance table for alcohol reduction process

Source	Sum of squares	df	Mean es	F value	P- value prob>F
Model	4681.43	9	520.16	118.87	<0.0001
A- Time	208.66	1	208.66	47.68	<0.0001
B- Temperature	266.96	1	266.96	61.01	<0.0001
C- Stirring speed	2162.16	1	2162.16	494.10	<0.0001
AB	756.06	1	756.06	172.78	<0.0001
AC	123.72	1	123.72	28.27	0.0003
BC	35.70	1	35.70	8.16	0.0171
A^2	35.77	1	35.77	8.17	0.0170
B^2	64.78	1	64.78	14.80	0.0032
C^2	817.70	1	817.70	186.86	<0.0001
Residual	43.76	10	4.38		
Lack of fit	16.12	5	3.22	0.58	0.7158
Pure error	27.64	5	5.53		
Cor Total	4725.19	19			

The model equation was presented both in actual and coded form. Equations 1 and 2 show the model equations both in coded and actual forms respectively. Model prediction can only be done with the coded form because the actual form has been scaled to accommodate their different units.

Final Equation in Terms of Coded Factors:

$$\text{Alcohol removed (\%)} = +40.72-3.61A+4.08B-11.62C-9.72AB-3.93AC+2.11BC-1.19A^2-1.61B^2+5.70C^2 \qquad (1)$$

Final Equation in Terms of Actual Factors:

$$\text{Alcohol removed (\%)} =-417.39768+195.77014\text{Time (hrs)} + 14.40753\text{Temperature (oC)} - 0.86158\text{ Stirring speed (Rpm)}-3.88860\text{Time (hrs)Temperature (oC)} - 0.15730\text{Time (hrs)Stirring speed (Rpm)}+8.45000E-003\text{ Temperature (oC) Stirring speed (Rpm)}-4.77073\text{Time (hrs)}^2 - 0.064207\text{Temperature(oC)}^2+2.28113E-003\text{Stirringspeed(Rpm)}^2 \qquad (2)$$

3.5. Residulas Diagnosis For Alcohol Reduction Process

Table 6 presents residuals generated for the alcohol reduction process. It was calculated as the differences between the actual values obtained from the experiments and the values predicted by the model. The assumptions made by ANOVA were assessed based of the behaviour of the residuals. Normal plot of residuals, residuals versus predicted plot, residuals versus run, and plots of predicted versus actual values were used to assess the behaviour of the residuals. ANOVA has some assumptions it made based on each plot. ANOVA assumed that the residuals should have constant variance and evenly distributed. The behaviour of each plot will show if the assumptions of the ANOVA were met. Some of the explanations to each plot are as follows;

The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line. Expect some moderate scatter even with normal data. Look only for definite patterns like an "S-shaped" curve, which indicates that a transformation of the response may provide a better analysis. Residual versus predicted plot is a plot of the residuals versus the ascending predicted response values. It tests the assumption of constant variance. The plot should be a random scatter (constant range of residuals across the graph.) Expanding variance ("megaphone pattern <") in this plot indicates the need for a transformation.

Residual versus run is a plot of the residuals versus the experimental run order. It allows you to check for lurking variables that may have influenced the response during the experiment. The plot should show a random scatter. Trends indicate a time-related variable lurking in the background. Blocking and randomization provide insurance against trends ruining the analysis. A graph of the actual response values versus the predicted response values. It helps you to detect a value, or group of values, that are not easily predicted by the model. The data points should be split evenly by the 45 degree line. The residuals plots are shown in Fig 1-4.

Table 6. Residuals generated for alcohol reduction process

Standard Order	**Actual Values**	**Predicted Values**	**Residual Values**
1	42.76	43.24	-0.48
2	64.84	63.32	1.52
3	66.28	66.62	-0.34
4	48.65	47.82	0.83
5	23.94	23.63	0.32
6	29.47	27.98	1.49
7	55.09	55.46	-0.38
8	22.56	20.93	1.63
9	44.18	43.17	1.01
10	26.57	28.73	-2.16
11	25.28	26.13	-0.85
12	42.17	42.47	-0.30
13	86.59	86.78	-0.19
14	39.33	40.28	-0.95
15	36.88	40.72	-3.84
16	39.53	40.72	-1.19
17	41.61	40.72	0.89
18	40.30	40.72	-0.42
19	40.86	40.72	0.14
20	43.99	40.72	3.27

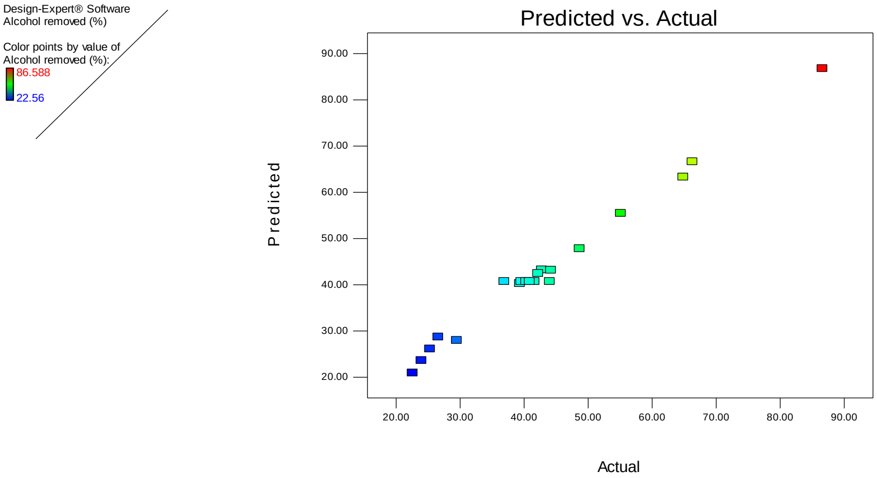

Fig.1. Plot of predicted versus actual values for the alcohol reduction process

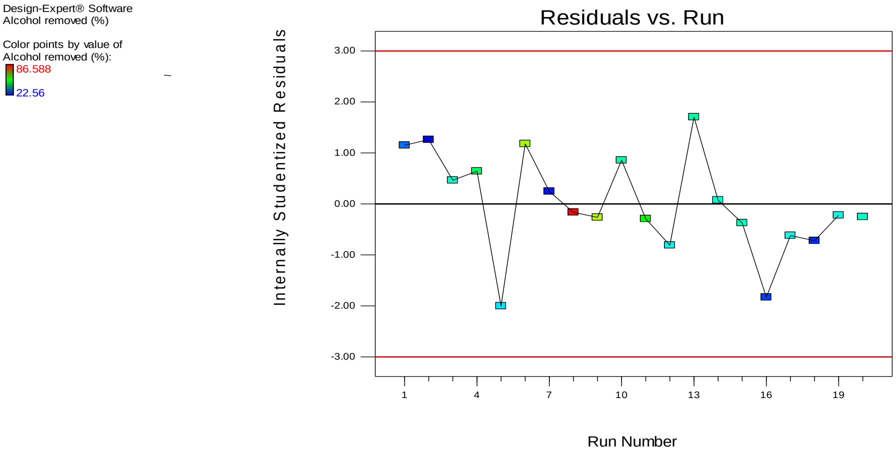

Fig. 2. Plot of residuals versus run order for the alcohol reduction process

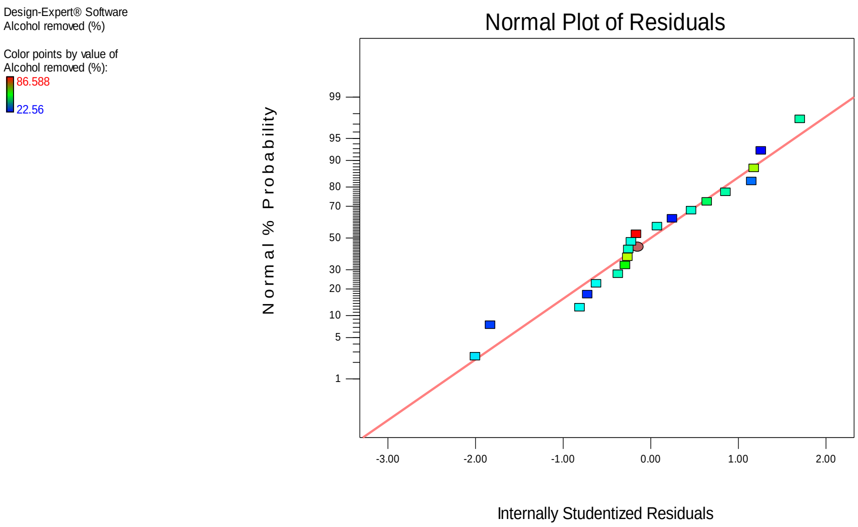

Fig 3. Normal probability plot for alcohol reduction process

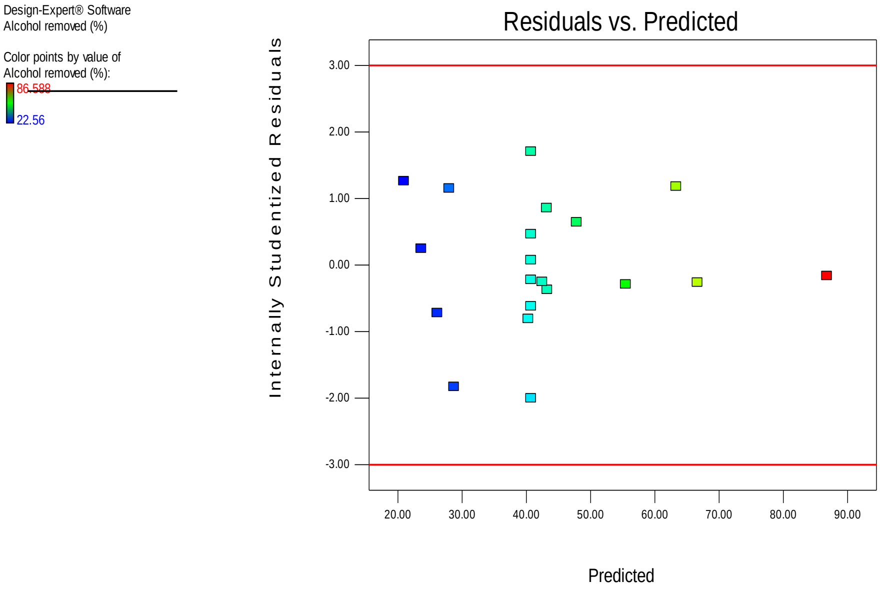

Fig.4. Plot of residuals versus predicted values

From the diagnostic plots (Fig 1-4), it can be seen that the plots met the assumptions of ANOVA which shows that the model equations generated can be used to predict the alcohol reduction of palm wine with higher accuracy.

3.6. 3D surface plots for the alcohol reduction process of palm wine

3D surface plot is very necessary for graphical interpretation of the interaction effects. Such three-dimensional surfaces can provide useful information about the behaviour of the system within the experiment design, facilitate an examination of the effects of the experimental factors on the responses and contour plots between the factors [19, 20, 21]. Figures 5-6 show the 3D surface plots with embedded contour plots of the interaction effect of time and temperature, and time and stirring speed. From figure 5, it can be seen that an increase in temperature from 35°C to 45°C increased the alcohol reduction at lower time interval, but at higher time interval increasing the temperature decreased the alcohol reduced. This can be attributed to the fact that increase in temperature increased the rate of volatilization of the alcohol from the palm wine at lower time interval but the reduction recorded at higher time interval may be due to the destruction of the membrane pore walls which affected the escape of the alcohol from the surface probably because the membrane was labelled as heat sensitive.

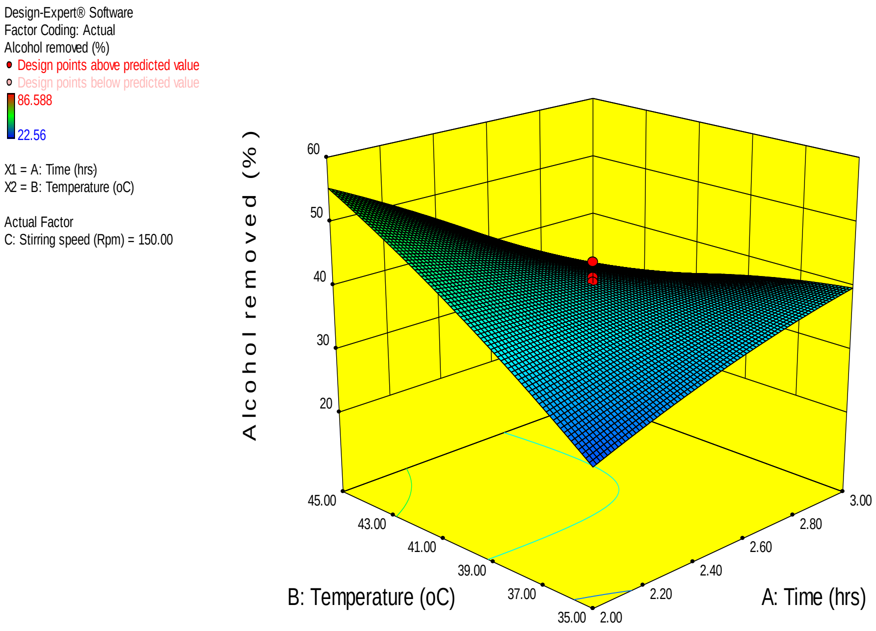

Fig. 5.3 D surface plot of temperature interaction with time for alcohol reduction process

Figure 6 shows the interaction effect of stirring speed and time. As can be seen from the plot, time did not have any effect on the alcohol reduced at lower stirring speed, but at higher stirring speed it was observed that increase in time decreased the alcohol reduced. Prolonged stirring speed resulted to serious agitation of the sample and it mounted a lot of pressure on the membrane surface. Since the sample was in direct contact with the membrane, it tends to disrupt the surface of the membrane there by reducing the transfer rate of the alcohol to the water side.

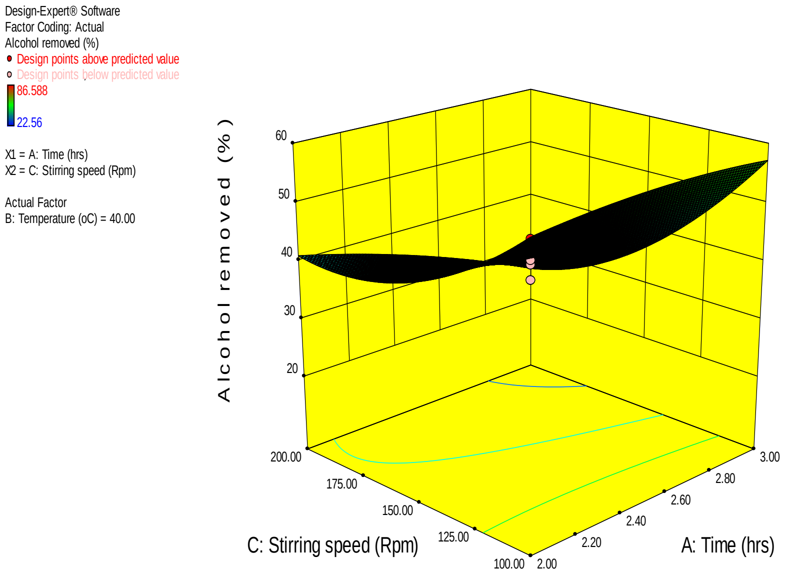

Fig. 6. 3 D surface plot of stirring speed interaction effect with time for alcohol reduction process

3.7. Optimal Conditions For Alcohol Reduction Process Of Palm Wine

Maximization optimization was done since our aim is to reduce the alcohol content of the palm wine. The optimization was done numerically and the optimum conditions were selected based on the highest desirability. The optimum conditions with their validations are shown on table 6. It can be seen that model predicted 55.2% reduction of alcohol using the conditions on the table. Initial alcohol content of the palm wine prior to dealcoholisation was 25%, which shows that after the dealcoholisation, that the amount of alcohol reduced to 11.2%. Based on the experimental values of 55.3%, it can be seen that the deviation from the predicted value was 0.18%. This shows that the model equation generated can predict the process well.

Table 6. Optimum condition and its validation for alcohol reduction process of palm wine.

			Alcohol reduced (%)		
Time (Hrs)	Temperature (^{o}C)	Stirring speed (Rpm)	Predicted Value	Experimental Value	Error (%)
3.00	45.00	153.08	55.2	55.3	0.18

IV. CONCLUSION

It can be concluded from this work that the alcohol content of palm wine can be trimmed down using osmotic membrane distillation method. The flavour and the aroma content of the wine will not be affected when polytetrafluoroethylene membrane is used. The palm wine was allowed to ferment for two days to enable the aroma and flavour contents to fully mature and optimizing with central composite design (CCD), the alcohol content which was initially 25% was trimmed down to 11.2%. Time, temperature and stirring speed were considered as the factors while percentage alcohol removal was the response. Quadratic model was generated for the alcohol reduction process and it was validated. It was observed that the single effect of the factors, interaction effect and quadratic effects were significant. The optimum conditions generated were validated with little error of 0.18%

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