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(RESEARCH ARTICLE)

Optimization of edible oil quality through vacuum bleaching: An experimental design and analysis study

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Abstract

A leading cooking oil producer in Zimbabwe encountered challenges in meeting quality specifications for edible oil, particularly due to high colour and free fatty acid levels. This prompted the exploration of vacuum bleaching, as the bleaching stage significantly impacts these parameters. Controlled experiments demonstrated that vacuum bleaching effectively reduces both colour intensity and free fatty acids, achieving optimal quality for soybean oil production while minimizing bleaching earth usage to no more than 12.5 kg. Predictive modelling with Minitab software guided the system's design, resulting in a highly efficient and reliable vacuum bleaching process that meets industry standards. It was concluded that the system performs more than adequately in lowering FFA and reducing colour to within specifications.

Keywords: Vacuum-Bleaching; Edible oil quality; Free fatty acid level; Process modelling; Process optimization

1. Introduction

Edible oil refining at the production plant typically allows for losses of no more than 3%, yet these losses often exceed this due to additional water washes and caustic waste, which retain some oil in the wash water. The bleaching process plays a critical role in refining, acting as the "safety net" for maintaining oil quality [1]. However, when processing local crude soybean oil or cottonseed oil, the pigment levels remain high, often reaching 50-70R on the colour scale, even after bleaching [2]. This requires reprocessing, extending cycle times, increasing production costs, and consuming excessive bleaching earth. Consequently, the oil's colour often exceeds the target specification of 3.5R, impacting quality and customer appeal, as consumers prefer lighter, visually appealing oils in yellow or gold hues [3].

Due to these challenges, the refinery's market competitiveness suffers, with darker oil less likely to be purchased compared to aesthetically pleasing alternatives. This study aims to develop a method for refining oil that minimizes losses and reduces raw material usage. Implementing a Vacuum Bleaching system is expected to improve colour quality and reduce energy consumption, directly enhancing refinery sales and profitability.

The aim of this research subsequently is to design a vacuum bleaching system based on the results of the analysis of experiments using MiniTab from samples of soybean seed oil (neutralised) that were collected from the refinery. The research objectives included performing experiments and gathering data collected from the log sheets of the refinery which will guide the methodology. After the experiments, the results helped design the vacuum system as a whole from the inlet subsystem, vacuum bleaching unit and outlet subsystem. The results were analysed under varying conditions which aided in the design of the system.

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2. Summary of literature

There are various parameters in manufacturing which constantly change and require companies to be agile [4]. Edible oil manufacturing requires operation at optimal conditions to achieve the required quality of oil. One of the methods that have been used is process optimization by using a comparative approach [5], although applied in machining parameters, it can be applied in oil production. Bleaching is a crucial step in the refining of edible oils, aimed at enhancing the oil's colour, stability, and overall quality for human consumption. It typically follows degumming and neutralization processes, acting as a "safety net" to remove impurities that could not be eliminated in previous refining stages [6]. Essential for edible oil refinement, bleaching removes peroxides, trace metals, and colour contaminants such as phosphatide pigments, chlorophyll, and residual soaps. The types of fats and oils that commonly undergo bleaching include beef tallow, palm oil, and sunflower seed oil, each presenting unique colour compounds like β-carotenoids, chlorophyll, and xanthophylls that impact their quality ([1]; [6]). The process's objective is to yield an oil low in impurities, meeting specific consumer requirements, including low phosphorus and peroxide values that extend shelf life and suitability for various edible applications [7].

The efficiency of bleaching depends on the clay used, with bentonite, attapulgite, and sepiolite being prominent due to their high absorbency and porosity. Bentonite, often acid-activated to improve its surface area, is particularly effective, though it requires optimization to avoid increasing free fatty acids in the final product. Acidic clay activation and moisture management are vital, as excessive acid can lead to higher free fatty acid content, reducing the oil's quality [1]. Moreover, vacuum bleaching at reduced pressures (50-125 mmHg) helps enhance the removal of chlorophyll and phospholipids, achieving a lower peroxide value and improving stability by minimizing oil-air interaction, ultimately leading to longer oil preservation [8].

The experimental approach has been used in various manufacturing processes to determine the optimum operation conditions as confirmed by [9] who confirmed the use of experimental analysis for process optimization using Taguchi experiments. This can also be successfully used in oil production. Process variables, including temperature, contact time, and moisture content, significantly affect bleaching outcomes. For example, temperatures between 90 to 125 °C improve adsorption efficiency and viscosity, enhancing clay-oil interaction. While higher temperatures improve chlorophyll removal, they must be controlled to prevent detrimental oxidation. Contact time within 15-45 minutes is optimal, with longer times potentially darkening the oil due to excessive oxidation [10]. Similarly, maintaining moisture within an optimal range supports the adsorption of chlorophyll and phospholipids, essential for colour stabilization and quality maintenance [1]. Modern techniques like microwave-assisted bleaching and nanotechnology have further advanced the process, with microwave heating reducing clay and time requirements, while nanoscale clay particles increase surface area for improved contaminant removal without compromising oil integrity ([7]; [6]).

Recent innovations such as the use of activated carbon from neem and tea leaves and nanotechnology have expanded options for sustainable and efficient bleaching processes. Activated carbon derived from natural sources demonstrates high adsorptive capacity and cost-effectiveness while reducing environmental impact, making it a promising alternative to traditional bleaching clays [11]. Nanotechnology, by reducing clay particle size, has been shown to enhance surface area and improve contaminant adsorption, leading to superior oil quality and bleaching efficiency [6]. Together, these advancements underscore a trend toward optimizing edible oil bleaching for higher quality, sustainability, and cost-effectiveness in the oil refining industry [12].

3. Methodology

The research methods are outlined below:

Table 1 Research Methodology

Objective	**Methodology**	**Tools**
To perform experiments	Experimental analysis	MiniTab software
To design the inlet, bleaching and discharge subsystem	System Design	Content analysis, Inventor
To Analyse and conduct predictions	Results and Analysis	MiniTab software

The Taguchi method of experiments was used for designing the experiments. A two-level system of high and low was used due to the availability of resources. The minimum number of required experiments was 12 with a total of 6 input parameters (factors) which were taken into consideration these include oil quantity, contact time, oil temperature (during bleaching), stirrer speed, earth quantity and vacuum level. The outputs included oil colour, FFA, soap content and absorbance. During the experiments, safety precautions were taken as emphasized by [13] to ensure there was no contamination of the samples and no possible accidents occurred.

The ranges of values for the input parameters were as follows as influenced by the preliminary experiments.

Table 2 Parametric Levels

Parameter	**Range**	
	low	**high**
Oil Quantity/g	100-200	300-400
Temperature	60-80	90-110
Contact time/min	5, 10	15-17
Stirrer Speed/rpm	100	1000
Earth Quantity/g	0.250-0.330	0.340-0.5
Vacuum /Hg	10	30

Using the L12 orthogonal array design, the parameters were varied as shown in the table below:

Table 3 Experimental Parameters

Oil Quantity	**Contact Time**	**Oil Temperature**	**Stirrer Speed**	**Earth Quantity**	**Vacuum Level**	**Experiment**
110.02	5	61.5	100	0.2683	10	1
118.2251	10	101	100	0.3633	30	2
106.366	8	106	1000	0.3791	10	3
103.3273	15	66	1000	0.4026	10	4
109.0343	17	102	100	0.477	30	5
103.9704	15	100	1000	0.2737	30	6
310.6015	10	101.5	1000	0.3001	10	7
311.3994	8	110	100	0.3622	30	8
315.0376	10	68	1000	0.3571	30	9
314.2159	15	96	100	0.2524	10	10
311.5836	15	72	1000	0.3266	30	11
301.9821	15	63	100	0.4148	10	12

For the procedure, an organic solvent (hexane) was added to 1ml of each sample from the 12 experiments and filled to the mark in 10ml volumetric flasks. The samples prepared are as shown below in Figure 1

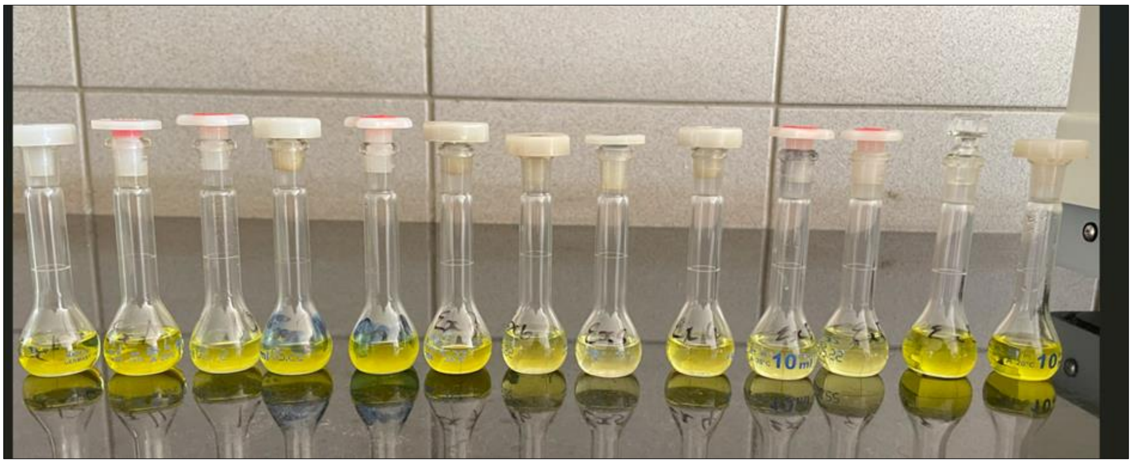

Figure 1 UV-Spec Samples for Experiments

The colours for the samples were analysed to see their colour spectra and the results showed that the sample colours were out of the recommended samples. The spectra is shown in Figure 2 below.

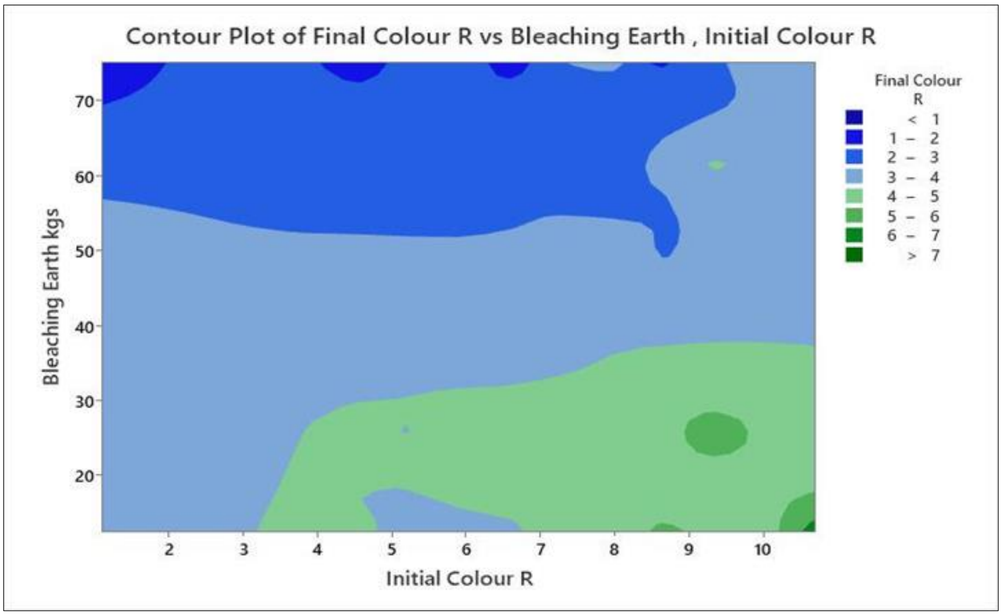

Figure 2 Data Collection Colour Spectra

4. Results and Discussion

The results from these experiments were as follows in Table 4: Results of Experiments, with the absorbance being determined using absorbance species (A) and transparency (T) of the sample. The results of the experiments are shown in table 4 below.

Table 4 Results of Experiments

Output	Colour / R	FFA/%	SOAP/ ppm	Absorbances A and T values		FFA mass/g	SOAP mass/ g
ref	7	0.16	54.72	0.551	28.1	14.0158	50.036
1	3.9	0.095	24.32	0.484	32.8	14.027	50.043
2	5.6	0.102	36.48	0.249	56.3	14.02	50.325
3	5.3	0.107	30.4	0.235	58.2	14.0407	50.183
4	5.9	0.093	18.24	0.331	46.6	14.48	50.058
5	4.6	0.104	30.4	0.202	62.8	14.0234	50.286
6	6.3	0.1	42.56	0.327	47	14.0509	50.103
7	7.2	0.1	48.64	0.597	25.3	14.0252	50.181
8	8.3	0.104	24.32	0.531	29.3	14.1278	50.169
9	7	0.094	48.64	0.572	26.8	14.0691	50.099
10	5	0.117	12.16	0.479	33.1	14.0134	25.048
11	6.2	0.086	30.4	0.683	20.8	14.0417	50.004
12	5.8	0.1	18.24	0.54	28.8	14.091	25.008

Regression equations which focused on the colour, FFA and soap content equations were derived using MiniTab. These equations were used as guidelines for the parameter specifications. Predictions were made on the colour, FFA and soap content as shown below.

Table 5 Parameter predictions

	Fit	SE Fit	95% CI	95% PI
Colour Predictions	3.48846	0.792011	(1.45253, 5.52439)	(0.355234, 6.62169)
FFA Predictions	0.0945897	0.0044479	(0.0831562, 0.106023)	(0.0781457, 0.111034)
Soap Content Predictions	14.7323	7.98714	(-5.79929, 35.2639)	(-16.8651, 46.3298)

The parameters were tested to see how they affected the colour of the oil. The steeper the gradient, the more the correlation between the colour and the process parameter, operating on the basis of the lower the colour value the better. From Figure 3 below, it is seen that oil quantity, stirrer speed, vacuum level, and earth quantity positively affect the colour of the oil. The lower the stirrer speed the lower the colour, the lower the vacuum level results in a better colour and the lower the oil quantity the better the resultant colour (batch production required). Also, it is seen that the lower the colour the longer the contact time (15.3 minutes on the average taken), For the test run, the lower the bleaching earth and oil temperature the better with averages of 0.28g and 66.1 degrees Celsius. The results are shown in Figure 3.

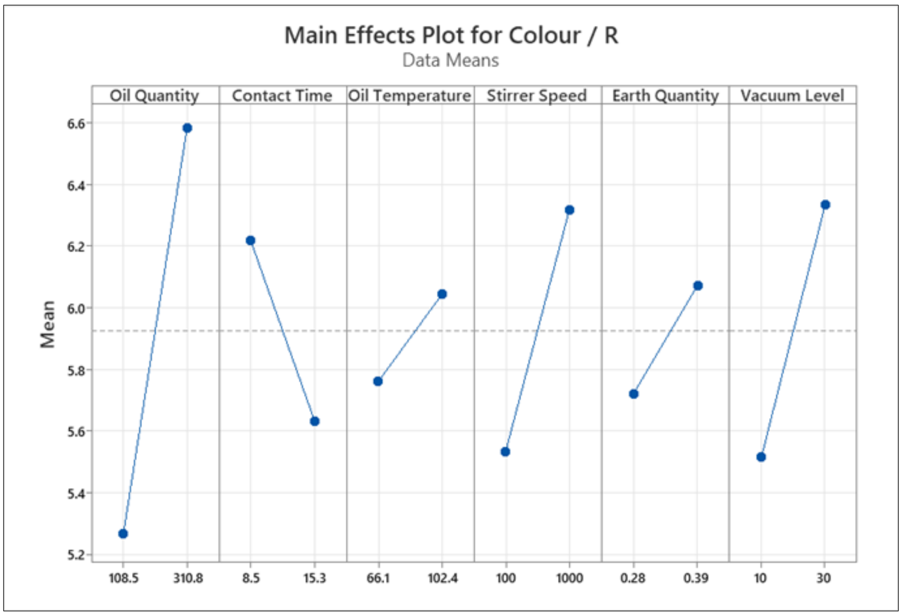

Figure 3 Main effects plot for colour

The next main effect plot to be considered is that of FFA against the process variables as shown below. From Figure 4 below, there is no gradient for oil quantity meaning that oil quantity does not have a bearing on the FFA percentage. Contact time does have a slight negative gradient implying that the FFA reduces as desired when contact time is increased. The earth quantity behaves as explained for the colour main effect plot. The notable parameters which have a significant bearing on FFA are oil temperature, vacuum level and stirrer speed. Oil temperature has the steepest gradient meaning that the FFA is sensitive to the temperature and the lower FFA results when temperature is low (66.1 degrees Celsius). This is closely followed by stirrer speed which shows that the higher the speed the lower the FFA. Vacuum level at 30Hg results in the lowest FFA reduction.

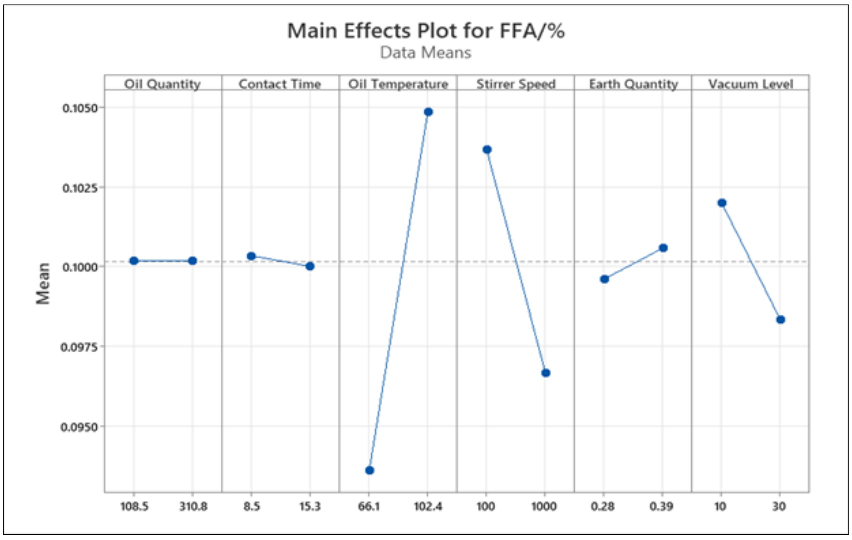

Figure 4 Main Effect Plot for FFA

The graph of the main effects plot for soap content against the six process variables is shown in Figure 5 below. It may be observed that oil quantity does not affect the soap present in the bleached oil. Stirrer speed, vacuum and contact time have the steepest gradients (in that order) showing that soap content is greatly affected by these (behave similar to that of colour). The stirrer speed at a low-value results in a value of below 25ppm. The lowest soap value was produced at 10Hg, meaning the lower the vacuum the better. The rest of the parameters behave similarly to colour.

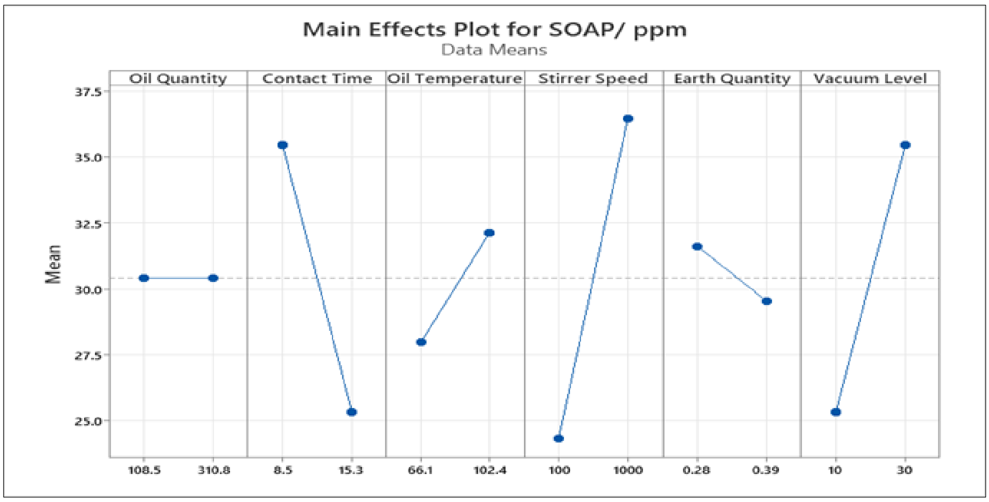

Figure 5 Main Effect Plot for Soap

All output parameters were analysed, namely of interest is colour, FFA and soap content. The value of interest is the p-value, in determining the degree to which an input parameter affects an output parameter, which should be below p=0.05 which indicates a strong correlation. Another value of interest is the R-sq. value which ideally should be as close to 1 as possible. The first table in each section will give the values of the two-level system and the values for each input parameter for the respective level.

Table 6 below shows the colour variance analysis. Values below 0.05 for the p-value are analysed which indicates a strong correlation with colour. Also, the R-sq. value is at 72.94% which shows that the regression explains 72.94% of the data well.

Table 6 Colour Variance Analysis

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Oil Quantity	1	6.3755	6.3755	7.43	0.042
Contact Time	1	0.4176	0.4176	0.49	0.517
Oil Temperature	1	0.7754	0.7754	0.90	0.386
Stirrer Speed	1	2.6726	2.6726	3.11	0.138
Earth Quantity	1	0.9728	0.9728	1.13	0.336
Vacuum Level	1	1.0726	1.0726	1.25	0.314
Error	5	4.2919	0.8584		
Total	11	15.8625			

Table 7 shows the FFA variance analysis. The p-value parameter below 0.05 is the oil temperature. Thus, oil temperature affects FFA to a greater extent. The r-sq. value in Table 5 7 84.45% meaning that the regression is 84.45% true for the set of data. The closer the value is to 1, the better and more accurate the regression.

Table 7 FFA Variance Analysis

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Oil Quantity	1	0.000017	0.000017	0.80	0.412
Contact Time	1	0.000013	0.000013	0.60	0.475
Oil Temperature	1	0.000386	0.000386	18.26	0.008

Stirrer Speed	1	0.000057	0.000057	2.71	0.160
Earth Quantity	1	0.000014	0.000014	0.68	0.448
Vacuum Level	1	0.000105	0.000105	4.95	0.077
Error	5	0.000106	0.000021		
Total	11	0.000680			

The data in Table 8, there is no factor with a p-value less than 0.05 and hence, no factor affects soap content to a significant extent.

Table 8 Soap Variance Analysis

Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**
Oil Quantity	1	0.07	0.068	0.00	0.979
Contact Time	1	294.92	294.919	3.38	0.125
Oil Temperature	1	16.65	16.649	0.19	0.681
Stirrer Speed	1	401.42	401.417	4.60	0.085
Earth Quantity	1	30.96	30.958	0.35	0.577
Vacuum Level	1	294.92	294.919	3.38	0.125
Error	5	436.49	87.298		
Total	11	1552.59			

The interaction plots are graphical representations of how input parameters interact with one another [9], in this case on a two-level system of high and low input parameters. This will be shown visually with the intersection of the line graphs. This intersection means that the two parameters at play are dependent on each other.

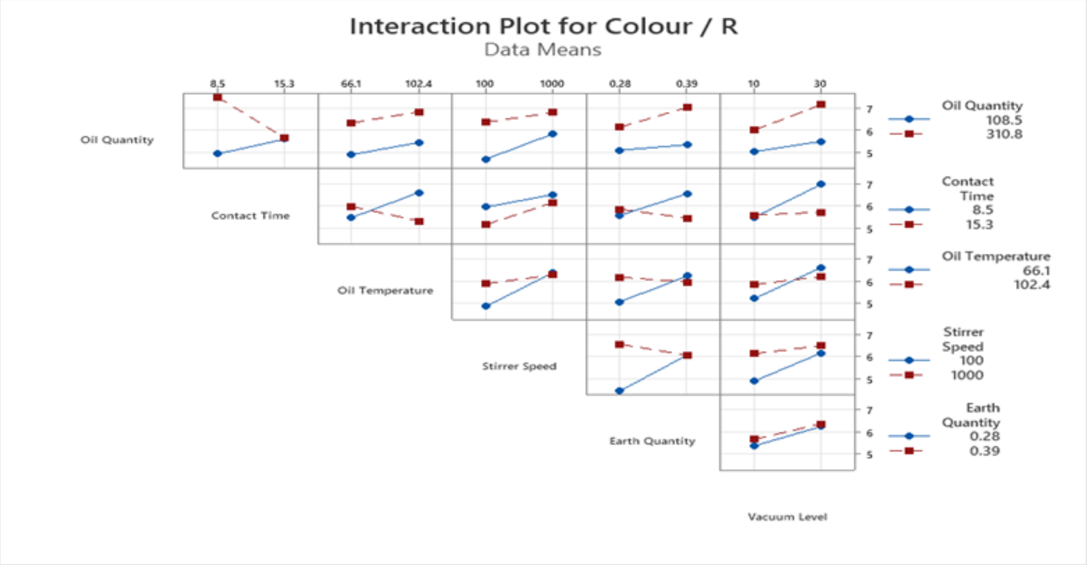

Figure 6 Colour Interaction Plot

The figure above, where the two graphs are parallel or almost parallel shows that the two input parameters under consideration are independent of each other. Consider the interaction of oil quantity and contact time, where the lower oil quantity and contact time result in a lower colour. Next is the first graph on the second row of contact time and oil temperature where the higher the oil temperature and contact time results in a lower colour. Also, contact time and earth quantity have a bearing on each other, with a reduction in both parameters resulting in a lower colour. Furthermore, on the same row, consider vacuum level and contact time with a reduction in both parameters resulting in a lower colour. In the third row for oil temperature and stirrer speed, a low temperature with a low stirrer speed will give lower colours. Oil temperature and earth quantity at low levels result in a lower colour. The lower the vacuum level and temperature of oil, the greater the colour reduction (also applies to stirrer speed and earth quantity).

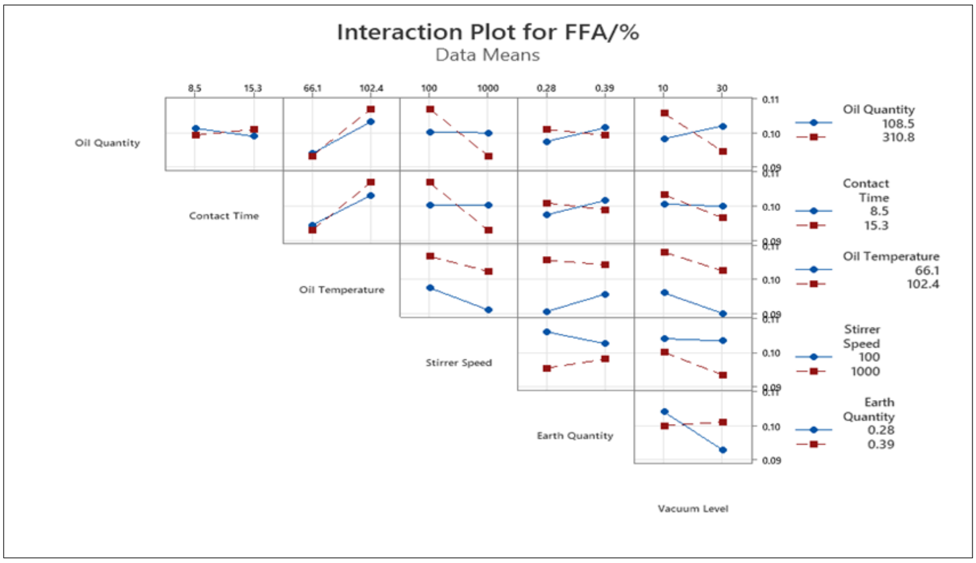

Figure 7 FFA Interaction Plot

Considering the first row with oil quantity and contact time the low oil quantity with a high contact time will reduce the FFA. Oil quantity and stirrer speed behave in such a manner that both high levels result in reduced FFA. Earth and oil quantity both on low levels result in a lowered FFA content. Also, vacuum level and oil quantity both on high levels result in reduced FFA. In the second row, contact time on a high with oil temperature on low reduces the FFA to specifications. Also, looking at the graph of stirrer speed vs contact time, it is observed that both parameters on a high level result in a reduction in FFA. A low contact time and earth quantity result in a reduction in FFA as well. For vacuum level and contact time on a high level result in lowered FFA. Lastly, earth quantity on low and vacuum level on high result in a significantly low value of FFA.

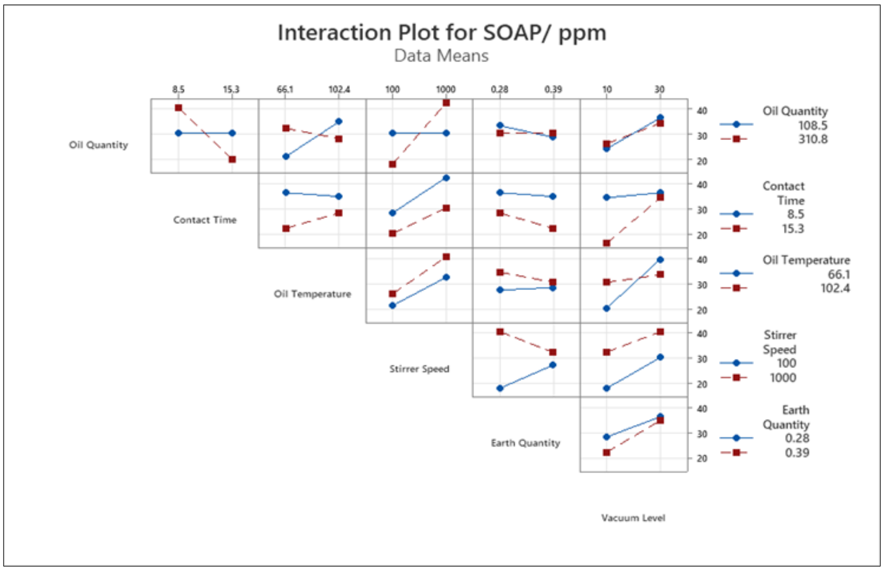

Figure 8 Soap Content Interaction Plot

Similarly, looking at the first row with oil quantity and contact time; the higher the quantity and contact time results in a significantly low soap content. Likewise, looking at oil quantity and temperature on a low factor for both parameters results in a lowered soap value. Also, stirrer speed and oil quantity interact in such a manner that a high oil quantity and a low stirrer speed result in a lowered soap value. Furthermore, for earth (high) and oil quantity (low) results in a slight soap reduction. Vacuum level and oil quantity interact in such a way that when both parameters are low, then a considerable reduction in soap content is realised. Lastly, looking at the third row of the collection of graphs, looking at oil temperature and vacuum level, low levels of both parameters result in the soap content reducing significantly.

5. Conclusion

In conclusion, it may be seen that the system performs more than adequately in lowering FFA and reducing colour to within specifications from the experimental analysis documentation. The developed system has been designed in such a way that it may be easily incorporated with an existing set-up for easy flow to a deodorisation system. Key recommendations indicate areas for possible improvement on the system which is dependent on the capital available for the installation and implementation of the vacuum bleaching system. With all projects, the initial start-up/investment costs will be significant. However, the evidence provided in the document presents numerous benefits which, in the long run, offset the initial investment.

When trying to find the break-even point for having a high or low level for contact time, it is recommended to focus on FFA reduction as deodorisation then reduces the colour but increases FFA. The factors that should be considered when applying process capability to a process include tooling, materials, production volume and production rate, machine condition, accuracy and repeatability of measurements.

Vacuum filtration after bleaching may be used for consistency with the system. However, vacuum filtration is expensive and will depend on the availability of capital for implementation. Including an absorbance check to test the effectiveness of bleaching based on the absorbance species present may be used as a method of quality control. Within the specifications are production capacities which should be adhered to and machines serviced within time. A PLC may be included to better tailor the system

Compliance with ethical standards

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Disclosure of conflict of interest

No conflict of interest to be disclosed.

Statement of consent

Informed consent was obtained during experiments for data collection.

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