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Research Article

Optimization of Raw Material, Decolourizing Earth and Temperature Use in the Decolourization of Palm Oil

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Abstract

The objective of this study was to evaluate the influence of raw material, decolourizing earth and temperature on the colour of bleached palm oil. Two types of decolourizing earth (American and Indian) were used. A four-factor centered composite response surface design was used to determine the effects of the different mentioned factors on the colour response of bleached palm oil at two DOBIs (2.3 and 1.3). The results obtained indicate that Indian earth with DOBI 2.3 oil has the colour variation contour lines at the high level of 16.0 red and at low level of 15.3 red. The decrease in colour around 15.4 is influenced by the effect of opposite temperature levels. The increase in color depends on the bleaching earth used. The temperature influences the colour of the bleached oil depending on the raw material. The bleaching temperature with American earth and a DOBI 1.3 oil, when it is at its high level (120°C) and at its low level (110°C), gives a colour of 15.8 red and 17.6 red, respectively. The optimal discoloration conditions (18.57 red) of CPO palm oil ($P \leq 0.05$) are for American earth (with DOBI 1.3 oil): 92°C and 0.035% for temperature and percentage of phosphoric acid; 105°C and 0.6% for temperature and percentage of decolourizing earth. For Indian earth (with DOBI 2.3 oil), we have the optimum (18.66 red): 105°C and 0.035% for temperature and percentage of phosphoric acid; 118.5°C and 0.88% for temperature and percentage of decolourizing earth.

Keywords

Decolourizing Earth, Colour, Temperature, Palm Oil, Optimization, Experimental Design

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1. Introduction

Palm oil is rich in saturated fatty acids and therefore solid at room temperature [11, 4]. It tolerates high temperatures well [5], goes little rancid and gives softness to foods. For this reason, it is useful for many food preparations. This oil is a traditional ingredient in the countries where it is produced, particularly in Cameroon. It is then eaten raw. 80% of its uses go to food (margarines, prepared meals, brioches, cereals, biscuits, ice cream), 19% to non-food products derived from oleochemistry (cosmetics, soaps, lubricants, candles, pharmaceutical products) and for the remaining 1% for the production of an agrofuel, biodiesel [6, 12, 13]. It is also an export product. In this case, it is used refined and deodorized. In Cameroon, as in other producing and consuming countries, raw red palm oil is usually in competition with refined palm oil. This phenomenon is part of the transformations in food models, linked to urbanisation and changes in supply. Consumption surveys reveal a decrease in consumption proportional to the increase in household income in favor of refined oils [16]. Everything that led to the increase in processing industries in Cameroon. Furthermore, the annual deficit in palm oil was 160 thousand tonnes in 2022 [9]. To cope with the importation of these products, refining companies must be competitive and produce refined oil with stable colour. However, oil refining is a set of delicate operations which involves several parameters for its smooth running. These parameters, including raw material, inputs and temperature, require refining operators to carefully combine said parameters. Specially since it is difficult to assess the individual contribution of each of them on the non-conformity of the colour of the refined oil. Nkouam *et al*. (2017) studied the influence of decolourizing earth and temperature during palm oil bleaching [14]. But, it is known that the sources of supply of crude palm oil refining companies are varied. They range from local producers to industrial palm nut processing companies. It then also becomes important to study the behavior of the refining process depending on raw material and inputs used. The objective of this work is then to optimize the combined use of raw material, decolourizing earth and temperature on bleached palm oil colour.

2. Material and Methods

2.1. Plant Material

Crude palm oil (CPO: Crude Palm Oil) and bleached palm oil (BPO: Bleached Palm Oil) were provided by the AZUR S. A. factory located in the Yassa district (Douala, Cameroun).

2.2. Methods

2.2.1. Characteristics and Effectiveness of the Decolourizing Earths Used

The characteristics of the earths used are those studied by [14]. This is American earth and Indian earth as presented in table 1.

Table 1. *Characteristics of the different decolourizing earths.*

Parameters	Earth 1	Earth 2	Standard
Moisture (g of water/100g dry matter)	11.76±0.06^{a}	9.57±0.24^{b}	11% max
pH	7.22±0.02^{a}	8.12±0.02^{b}	6.5±1.0
Acidity	0.29±0.01^{a}	0.12'±0.01^{c}	0.3% max H_2SO_4

Values in rows with different superscript letters are significantly different ($p < 0.05$)
Earth 1 = American; Earth 2 = Indian

Furthermore, the aforementioned authors also found that the two earths presented efficiency values that were too far apart (table 2). The desired character being the reduction of color and the standard is 20 red max. However, American earth being the best, it will be used for poor quality crude oil (DOBI=1.3). Indian earth will be used for better quality crude oil (DOBI=2.3).

Table 2. *Effectiveness of decolourizing earths.*

Types of decolourizing earth	Red color of the BPO
Earth 1	15.13±0.15^{a}
Earth 2	15.78±0.49^{b}

Values in columns with different superscript letters are significantly different ($p < 0.05$)
Earth 1 = American; Earth 2 = Indian

2.2.2. Determination of DOBI and Bleaching of Crude Palm Oil

These two parameters were determined according to the methods described by [14]. Likewise, the characteristics of the inputs for carrying out bleaching are identical. We associated a DOBI oil equal to 1.3 (table 3). It should be remembered that the DOBI (Deterioration of Bleachability Index) is an indicator of the bleaching capacity of crude palm oil based on the quantity of carotenes present in the crude oil and the quantity of secondary oxidation metabolites. Good crude palm oil that is easily bleached will have a DOBI of 4, while average raw quality will have a DOBI of 2.5 à3 [8].

Table 3. *Sampling of palm oils.*

Inputs	Mass (g)	DOBI	FFA	Temperature (°C)	Percentage (%)
CPO	50	2.3 and 1.3	5.2		
H_3PO_4				100	0.05
Decolourizing earth				120	1.2

The color of the bleached and deodorized oil, as well as the standard of the oil refined at AZUR, did not change (table 4).

Table 4. *Standard of refined oil at AZUR S.A.*

Products	Standards
BPO	20 red max
RBD	3 red max
Oléine	4 red max

BPO: Bleached palm oil
RBD: Refined Bleached Deodorized oil

2.2.3. Evaluation of the Effect of Raw Material, Decolourizing Earth and Temperature on the Colour Variation of BPO

A four-factor composite experimental design was used to evaluate the effect of raw material, decolourizing earth and temperature on the colour variation of BPO. The bleaching temperatures (in ℃), the percentage of phosphoric acid which fixes the phosphatides of the oil (in %) and the percentages of decolourizing earth (in %) were used to determine the different colour responses.

(i). Choice of Factors and Experimental Domain

Bleaching temperatures (in ℃), phosphoric acid percentage (in %) and decolourizing earth percentages (in %) were used to determine the different colour responses. The literature of scientific documents and the ranges used in business made it possible to confirm the choice of factors (table 5).

Table 51. *Factors, domains of variation and centre of domain.*

Real variables		Coded variables					
		-α	-1	0	+1	+α	ΔXi
Introduction temperature of phosphoric acid (°C)	X_1	85	90	95	100	105	10
% phosphoric acid	X_2	0.035	0.050	0.065	0.080	0.095	0.030
Introduction temperature of decolourizing earth (°C)	X_3	105	110	115	120	125	10
% decolourizing earth	X_4	0.6	0.8	1.0	1.2	1.4	0.4
Red colour response	Y_i						

For an experimental design with n factors, the number of experiments is given by the formula: $N=2^n+2n+C$

Number of obligatory trials $2^n = 16$, with n=4; Number of star points 2n=8; Number of tests at centre C ≥ 4. For this study C=6.

We thus have N = 30 experiments, with $\alpha = (2^n)^{0.25}$, n=4 and α=2

This value of α is characteristic of the iso varying plan by rotation with 6 points at the centre. This gives an axial distance of 2 (Statgraphics centurion XVI (1982-2012)).

(ii). Choice of Experimental Responses

Based on the objective of this work, we chose the red colour response obtained after decolourization. The widely used Response Surface Method (RSM) transformation [2] to obtain real variables is as follows: $X = U_i + \Delta U_i \times x_i$

The previous equation represents the transformation of the coded values of the experiment matrix into real values of the experiment matrix where:

X: is the value of the natural variable (real value); *xi:* the coded variable value given by the matrix; *Ui:* the value of the centre of the range of the factor.

$$U_i = \frac{U_{max}+U_{min}}{2}$$

Ui is called «variation step» and characterizes the variation of the real variable associated with a variation of one unit of the corresponding coded variable.

(iii). Experiment Matrix

The experiment matrix (tables 6 and 7) is the table which presents all the experiments to be carried out with the levels of variation of each of the factors. This table is therefore composed of coded variables between +1, 0, -1, α and -α. The proposed model makes it possible to explain the mechanism of the phenomenon studied and to properly represent the experimental response studied in the chosen domain. The responses are regularly expressed as polynomials. Red colour after bleaching is the response. We applied the polynomial model of degree 2, for four variables. The model is of the following form:

$$Y_i = \beta_o + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j$$

where Yi is the predicted response, β_0 the constant, the linear coefficients βi, the square coefficients βii, the interaction coefficients βij. x_i, and x_i^2 are the levels of the independent variables.

Table 6. *Matrix of composite experimental design of American decolourizing earth.*

	Coded values				Real values				Responses
Nº	X_1	X_2	X_3	X_4	U_1	U_2	U_3	U_4	Y
1	0	0	+ α	0	95	0.065	125	1	YAde1
2	- α	0	0	0	85	0.065	115	1	YAde2
3	+ α	0	0	0	105	0.065	115	1	YAde3
4	+1	-1	+1	-1	100	0.05	120	0.8	YAde4
5	0	0	- α	0	95	0.065	105	1	YAde5
6	-1	+1	+1	+1	90	0.08	120	1.2	YAde6
7	-1	-1	+1	-1	90	0.05	120	0.8	YAde7
8	0	0	0	0	95	0.065	115	1	YAde8
9	0	0	0	0	95	0.065	115	1	YAde9
10	-1	-1	-1	+1	90	0.05	110	1.2	YAde10
11	+1	+1	-1	-1	100	0.08	110	0.8	YAde11
12	0	0	0	0	95	0.065	115	1	YAde12
13	0	+ α	0	0	95	0.095	115	1	YAde13
14	+1	+1	+1	+1	100	0.08	120	1.2	YAde14
15	0	0	0	+ α	95	0.065	115	1.4	YAde15
16	-1	+1	-1	+1	90	0.08	110	1.2	YAde16
17	+1	+1	+1	-1	100	0.08	120	0.8	YAde17
18	-1	+1	+1	-1	90	0.08	120	0.8	YAde18
19	0	0	0	- α	95	0.065	115	0.6	YAde19
20	-1	+1	-1	-1	90	0.08	110	0.8	YAde20
21	+1	-1	-1	-1	100	0.05	110	0.8	YAde21
22	-1	-1	+1	+1	90	0.05	120	1.2	YAde22

	Coded values				Real values				Responses
Nº	X_1	X_2	X_3	X_4	U_1	U_2	U_3	U_4	Y
23	+1	+1	-1	+1	100	0.08	110	1.2	YAde23
24	-1	-1	-1	-1	90	0.05	110	0.8	YAde24
25	0	0	0	0	95	0.065	115	1	YAde25
26	+1	-1	-1	+1	100	0.05	110	1.2	YAde26
27	0	0	0	0	95	0.065	115	1	YAde27
28	0	- α	0	0	95	0.035	115	1	YAde28
29	0	0	0	0	95	0.065	115	1	YAde29
30	+1	-1	+1	+1	100	0.05	120	1.2	YAde30

YAde: American decolourizing earth response

Table 7. Matrix of composite experimental design of Indian decolourizing earth.

	Coded values				Real values				Responses
Nº	X_1	X_2	X_3	X_4	U_1	U_2	U_3	U_4	Y
1	0	0	0	0	95	0.065	115	1	YIde1
2	0	0	0	0	95	0.065	115	1	YIde2
3	+1	-1	+1	-1	100	0.05	120	0.8	YIde3
4	0	+ α	0	0	95	0.095	115	1	YIde4
5	0	0	0	- α	95	0.065	115	0.6	YIde5
6	+ α	0	0	0	105	0.065	115	1	YIde6
7	-1	-1	-1	+1	90	0.05	110	1.2	YIde7
8	-1	-1	+1	-1	90	0.05	120	0.8	YIde8
9	+1	+1	-1	-1	100	0.08	110	0.8	YIde9
10	+1	+1	-1	+1	100	0.08	110	1.2	YIde10
11	0	0	- α	0	95	0.065	105	1	YIde11
12	0	0	0	+ α	95	0.065	115	1.4	YIde12
13	- α	0	0	0	85	0.065	115	1	YIde13
14	0	0	0	0	95	0.065	115	1	YIde14
15	-1	-1	-1	-1	90	0.05	110	0.8	YIde15
16	-1	+1	+1	+1	90	0.08	120	1.2	YIde16
17	-1	+1	-1	-1	90	0.08	110	0.8	YIde17
18	-1	+1	-1	+1	90	0.08	110	1.2	YIde18
19	0	0	0	0	95	0.065	115	1	YIde19
20	-1	+1	+1	-1	90	0.08	120	0.8	YIde20
21	+1	+1	+1	-1	100	0.08	120	0.8	YIde21
22	-1	-1	+1	+1	90	0.05	120	1.2	YIde22

	Coded values				Real values				Responses
Nº	X_1	X_2	X_3	X_4	U_1	U_2	U_3	U_4	Y
23	0	- α	0	0	95	0.035	115	1	YIde23
24	+1	-1	-1	-1	100	0.05	110	0.8	YIde24
25	+1	-1	+1	+1	100	0.05	120	1.2	YIde25
26	+1	-1	-1	+1	100	0.05	110	1.2	YIde26
27	0	0	0	0	95	0.065	115	1	YIde27
28	+1	+1	+1	+1	100	0.08	120	1.2	YIde28
29	0	0	+ α	0	95	0.065	125	1	YIde29
30	0	0	0	0	95	0.065	115	1	YIde30

YIde: Indian decolourizing earth response

(iv). Model Validation

Correlation coefficient (R^2), adjusted correlation coefficient (adjusted R^2), Absolute Mean Deviation Analysis (AADM), bias factor (Bf) and accuracy factor (Af) were used to check validation of the model and prediction of Y responses in the domain defined for the study. The correlation coefficient provides information on the average error of manipulations and must be greater than or equal to 0.90. This adjusted coefficient must be greater than or equal to 0.80. Furthermore, the Absolute Mean Deviation Analysis (AADM) indicates the absolute deviation between the experimental and calculated responses. Its value must be between 0 and 0.3, but should ideally tend towards 0. The bias and accuracy factors are ratios between the experimental and calculated responses. They are indicators of the error generated between the experimental and calculated responses. It should ideally tend towards 1, its value must be between 0.75 and 1.25 [2].

$$AADM = \sum_{i=1}^{P} \frac{\left(\left|\frac{Yiexp - Yical}{Yiexp}\right|\right)}{P}$$

With: Y_{iexp} the experimental response and Y_{ical} the response calculated from the model for an experiment i; p being the total number of experiments.

The bias factor is given by the formula $Bf = 10^B$ with $B = \frac{1}{p}\sum_{i=1}^{P} \log\left(\frac{Yical}{Yi\ exp}\right)$

The accuracy factor is given by the formula $Af = 10^A$ with $A = \frac{1}{p}\sum_{i=1}^{P} \log\left|\frac{Y_{ical}}{Y_{i\ exp}}\right|$

2.2.4. Data Analysis and Processing

Absolute Mean Deviation Analysis (AADM), bias and accuracy factors were determined using Excel 2013. Ink software. Calculation of the correlation coefficient (R^2) and adjusted correlation coefficient (adjusted R^2) was carried out using Statgraphics centurion XVI software (1982-2012) version 16.1.18, Statpoint Technologies, Inc. This software was also used to obtain results in the form of matrix operations (fitness line, effects diagram, model equations) and to assess the behavior of the colour response as a function of the levels of variation of the different variables materialized by the iso-response curves and the surface response 3-D curves.

3. Results and Discussion

3.1. Results of the Experimental Design

3.1.1. Experimental Design with American Decolourizing Earth

The test matrix, obtained from the experiment matrix with use of American decolourizing earth and palm oil with a DOBI 1.3 characteristic, gives the colour values for all combinations (table 8). It makes it possible to appreciate the variation in the colour of bleached palm oil depending on the different combinations of different variables. The combinations could be explained by the fact that the effectiveness of palm oil decolourization depends on the doses of the decolourizing earth and the temperature levels applied [14].

Table 8. *Matrix of tests with American decolourizing earth.*

Experiment design with American decolourizing earth						
Nº	**X_1(°C)**	**X_2(%H_3PO_4)**	**X_3(°C)**	**X_4(%Earth)**	**Y experimental**	**Y theoretical**
1	95	0.065	125	1	16.4	16.2542
2	85	0.065	115	1	16.3	16.1208
3	105	0.065	115	1	15.7	15.5375
4	100	0.05	120	0.8	16.3	16.3833
5	95	0.065	105	1	17	16.8042
6	90	0.08	120	1.2	15.8	15.9125
7	90	0.05	120	0.8	16.5	16.6625
8	95	0.065	115	1	17	16.8833
9	95	0.065	115	1	16.9	16.8833
10	90	0.05	110	1.2	16.8	16.9125
11	100	0.08	110	0.8	15.6	15.7333
12	95	0.065	115	1	17	16.8833
13	95	0.095	115	1	15.4	15.2042
14	100	0.08	120	1.2	15.8	15.8833
15	95	0.065	115	1.4	16.4	16.3042
16	90	0.08	110	1.2	15.7	15.75
17	100	0.08	120	0.8	15.8	15.8958
18	90	0.08	120	0.8	16	16.05
19	95	0.065	115	0.6	17.3	17.0542
20	90	0.08	110	0.8	16	16.1625
21	100	0.05	110	0.8	17	17.0958
22	90	0.05	120	1.2	16.2	16.2
23	100	0.08	110	1.2	15.4	15.4458
24	90	0.05	110	0.8	17.6	17.65
25	95	0.065	115	1	16.8	16.8833
26	100	0.05	110	1.2	16.4	16.4833
27	95	0.065	115	1	16.8	16.8833
28	95	0.035	115	1	17	16.8542
29	95	0.065	115	1	16.8	16.8833
30	100	0.05	120	1.2	16	16.0458

X_1: Introduction temperature of phosphoric acid; X_2: Percentage of phosphoric acide; X_3: Introduction temperature of decolourizing earth; X_4: Percentage of decolourizing earth

Equation 1 is obtained from the experimental design with the use of American decolourizing earth and a raw material with DOBI 1.3 characteristic.

$$\text{Colour} = 16.8833 - 0.145833X_1 - 0.4125X_2 - 0.1375X_3 - 0.1875X_4 - 0.263542\,X_1^2 + 0.03125X_1X_2 + 0.06875X_1X_3 +$$

$$0.03125X_1X_4 - 0.213542\,X_2^2 + 0.21875X_2X_3 + 0.08125X_2X_4 - 0.0885417\,X_3^2 + 0.06875X_3X_4 - 0.0510417\,X_4^2 \quad (1)$$

(i). Mathematical Analysis of the Results

We observe from the signs of the coefficients of the model (table 9) that six interactions namely X_1X_2 (introduction temperature of phosphoric acid/% phosphoric acid), X_1X_3 (introduction temperature of phosphoric acid/introduction temperature of decolourizing earth), X_1X_4 (introduction temperature of phosphoric acid/% decolourizing earth), X_2X_3 (% phosphoric acid/introduction temperature of decolourizing earth), X_2X_4 (% phosphoric acid/% decolourizing earth), X_3X_4 (introduction temperature of decolourizing earth/% decolourizing earth), with respectively coefficients 0.03125, 0.06875, 0.03125, 0.21875, 0.08125 and 0.06875 contribute to the increase in the colour of bleached palm oil instead of decreasing it. All others coefficients with negative signs contribute to the decrease in the colour of bleached palm oil. The most signifcant terms in palm oil decolourisation in descending order are: X_2: 38.95% (% phosphoric acid), X_1^2 : 15.90% (quadratic term; introduction temperature of phosphoric acid), X_2X_3: 10.95% (% phosphoric acid/ introduction temperature of decolourizing earth), X_2^2 : 10.44% (quadratic term; % phosphoric acid), X_4: 8.05% (% decolourizing earth), X_1: 4.87% (introduction temperature of phosphoric acid), X_3: 4.33% (introduction temperature of decolourizing earth). These five terms contribute 93.49% to the discolouration of palm oil during crude oil refining. The interactions X_4^2, X_1X_2, X_1X_4 which have insignificant weights on the response (0.60%: 0.22%, 0.22% respectively) must be taken away from the model equation.

Table 9. Coefficient of factors and weights on the response with American earth.

Factors	Coefficient	Squared coefficient	% effect
Constant	16.8833		
X_1	-0.145833	0.02126726	4.86837924
X_2	-0.4125	0.17015625	38.9511861
X_3	-0.1375	0.01890625	4.32790957
X_4	-0.1875	0.03515625	8.04776573
X_1^2	-0.263542	0.06945439	15.8990969
X_1X_2	0.03125	0.00097656	0.22354905
X_1X_3	0.06875	0.00472656	1.08197739
X_1X_4	0.03125	0.00097656	0.22354905
X_2^2	-0.213542	0.04560019	10.4385312
X_2X_3	0.21875	0.04785156	10.9539034
X_2X_4	0.08125	0.00660156	1.51119157
X_3^2	-0.0885417	0.00783963	1.79460343
X_3X_4	0.06875	0.00472656	1.08197739
X_4^2	-0.0510417	0.00260526	0.59637996
		$\sum \beta_i^2$ =0.43684485	

The equation of the reduced model after remouval of non-influential factors is as follows:

$$Colour = 16.8833-0.145833X_1-0.4125X_2-0.1375X_3-0.1875X_4 - 0.263542\,X_1^2 + 0.06875X_1X_3 - 0.213542\,X_2^2 + 0.21875X_2X_3 + 0.08125X_2X_4 - 0.0885417\,X_3^2 + 0.06875X_3X_4 \quad (2)$$

The optima in coded and real values are presented in Table 10.

Table 10. Optima in coded and real values of equation (2).

Factors	X_1	X_2	X_3	X_4	$Y_{optimum}$
Coded values	-0,56	-1,99	-2	-1,99	
Real values	92.2°C	0.035%	105°C	0.6%	18.5658

X_1: Introduction temperature of phosphoric acid; X_2: Percentage of phosphoric acide; X_3: Introduction temperature of decolourizing earth; X_4: Percentage of decolourizing earth

(ii). Graphical Analysis of Results and Validation of Models

Table 11. Validation of the decolouration model.

Validation elements	Obtained values	Standards values	Acceptable values
R^2	0.96	1	≥ 0.90
Ajusted R^2	0.92	1	≥0.80
AADM	0.00657329	0	0 – 0.3
Bf	1.000025194	1	0.75 – 1.25
Af	1.000025194	1	0.75 – 1.25

Statistical analysis showed that the proposed model is valid with satisfactory values of R^2, ajusted R^2, Bf and AADM. Indeed, the coefficient of the regression line is 0.96. The adjusted coefficient of determination is 0.92, and the bias and accuracy factors have a value of 1.000. Finally, the AADM value tends towards 0 (table 11). The model can be used to navigate within the experimental domain and predict the evolution of the colouring of bleached palm oil depending on the factors.

3.1.2. Experiment Design with Indian Decolourizing Earth

The test matrix, obtained from the experiment matrix with the use of Indian decolourizing earth and palm oil with a DOBI 2.3 characteristic, give the response values for all combinations (table 12).

Table 12. Matrix of tests with Indian decolourizing earth.

Experiment design with Indian decolourizing earth						
Nº	X_1 (°C)	X_2 (%H_3PO_4)	X_3 (°C)	X_4 (%Earth)	Y experimental	Y theoretical
1	95	0.065	115	1	16.4	16.4
2	95	0.065	115	1	16.4	16.4
3	100	0.05	120	0.8	17	17.0917
4	95	0.095	115	1	16.2	16.2458
5	95	0.065	115	0.6	16	15.7792
6	105	0.065	115	1	17	16.7792
7	90	0.05	110	1.2	14.5	14.1625
8	90	0.05	120	0.8	15.2	15.2958
9	100	0.08	110	0.8	15.6	15.4917
10	100	0.08	110	1.2	15.9	15.7458
11	95	0.065	105	1	15.2	15.3458

Experiment design with Indian decolourizing earth						
Nº	**X_1 (°C)**	**X_2 (%H_3PO_4)**	**X_3 (°C)**	**X_4 (%Earth)**	**Y experimental**	**Y theoretical**
12	95	0.065	115	1.4	15	15.0625
13	85	0.065	115	1	14.8	14.8625
14	95	0.065	115	1	16.4	16.4
15	90	0.05	110	0.8	15.3	15.3583
16	90	0.08	120	1.2	15.9	15.6625
17	90	0.08	110	0.8	16	15.9458
18	90	0.08	110	1.2	15.5	15.625
19	95	0.065	115	1	16.4	16.4
20	90	0.08	120	0.8	15.7	15.7583
21	100	0.08	120	0.8	15.4	15.6792
22	90	0.05	120	1.2	14	14.325
23	95	0.035	115	1	16.4	16.1958
24	100	0.05	110	0.8	16.6	16.7792
25	100	0.05	120	1.2	16.7	16.6958
26	100	0.05	110	1.2	16	16.1583
27	95	0.065	115	1	16.4	16.4
28	100	0.08	120	1.2	16	16.1583
29	95	0.065	125	1	16	15.6958
30	95	0.065	115	1	16.4	16.4

X_1: Introduction temperature of phosphoric acid; X_2: Percentage of phosphoric acide; X_3: Introduction temperature of decolourizing earth; X_4: Percentage of decolourizing earth

Equation 3 is obtained from the experimental design using Indian decolourizing earth and a raw material with a DOBI 2.3 characteristic.

$$Colour = 16.4 + 0.479167X_1 + 0.0125X_2 + 0.0875X_3 - 0.179167X_4 - 0.144792X_1^2 - 0.46875X_1X_2 + 0.09375X_1X_3 + 0.14375X_1X_4 - 0.0447917X_2^2 - 0.03125X_2X_3 + 0.21875X_2X_4 - 0.219792X_3^2 + 0.05625X_3X_4 - 0.244792X_4^2 \quad (3)$$

(i). Mathematical Analysis of the Results

We observe from the signs of the coefficients of the model (table 13) that three factors and four interactions which are: X_1 (introduction temperature of phosphoric acid), X_2 (% phosphoric acid), X_3 (introduction temperature of decolourizing earth), X_1X_3 (introduction temperature of phosphoric acid/introduction temperature of decolourizing earth), X_1X_4 (introduction temperature of phosphoric acid/% decolourizing earth), X_2X_4 (% phosphoric acid/% decolourizing earth) et X_3X_4 (introduction temperature of decolourizing earth/% decolourizing earth) with respectively coefficients 0.479167, 0.0125, 0.0875, 0.09375, 0.14375, 0.21875 and 0.05625 contribute to the increase in the colour of bleached palm oil instead of decreasing it. All other coefficients with negative signs contribute to the decrease in the colour of bleached palm oil. The most significant terms in palm oil decolourisation in descending order are: X_1: 32.71% (introduction temperature of phosphoric acid), X_1X_2: 31.31% (introduction temperature of phosphoric acid /% phosphoric acid), X_4^2 : 8.54% (quadratic term; % decolourizing earth), X_3^2 : 6.88% (quadratic term; introduction temperature of decolourizing earth), X_2X_4: 6.82% (% phosphoric acid/% decolourizing earth). These five terms contribute 86.25% to the discolouration of palm oil during crude oil refining. The factor X_2 (% phosphoric acid) and the interactions X_3X_4, X_2^2, X_2X_3 which have insignificant weights on the response (0.02%, 0.45%, 0.29%, 0.14% respectively) must be removed from the model equation.

Table 13. Coefficient of factors and weights on the response with Indian decolourizing earth.

Factors	Coefficient	Squared coefficient	% effect
Constant	16.4		
X_1	0.479167	0.22960101	32.7118756
X_2	0.0125	0.00015625	0.02226136
X_3	0.0875	0.00765625	1.09080658
X_4	-0.179167	0.03210081	4.57348952
X_1^2	-0.144792	0.02096472	2.98690066
X_1X_2	-0.46875	0.21972656	31.3050359
X_1X_3	0.09375	0.00878906	1.25220144
X_1X_4	0.14375	0.02066406	2.94406471
X_2^2	-0.0447917	0.0020063	0.28584246
X_2X_3	-0.03125	0.00097656	0.13913349
X_2X_4	0.21875	0.04785156	6.81754115
X_3^2	-0.219792	0.04830852	6.88264559
X_3X_4	0.05625	0.00316406	0.45079252
X_4^2	-0.244792	0.05992312	8.53740897
		$\sum \beta_i^2 = 0.70188887$	

The equation of the reduced model after elimination of non-influential factors is as follows:

$$Colour = 16.4 + 0.479167X_1 + 0.0875X_3 - 0.179167X_4 - 0.144792 X_1^2 - 0.46875X_1X_2 + 0.09375X_1X_3 + 0.14375X_1X_4 + 0.21875X_2X_4 - 0.219792 X_3^2 - 0.244792 X_4^2 \quad (4)$$

Solving this equation makes it possible to obtain the optima in coded and in real values (table 14).

Table 14. Optima in coded and real values of equation (4).

Factors	X_1	X_2	X_3	X_4	$Y_{optimum}$
Coded values	+2	-2	+0.71	-0.58	
Real values	105°C	0.035%	118.5°C	0.88%	18.6643

X_1: Introduction temperature of phosphoric acid; X_2: Percentage of phosphoric acide; X_3: Introduction temperature of decolourizing earth; X_4: Percentage of decolourizing earth

(ii). Graphical Analysis of Results and Validation of Model

We also observe that the proposed model is valid with satisfactory values of R^2, ajusted R^2, Bf and AADM. Indeed, the coefficient of the regression line is 0.95. The adjusted coefficient of determination is 0.90, and the bias and accuracy factors have a value of 1.000. Finally, the AADM value tends towards 0 (table 15). The model can be used to navigate within the experimental domain and predict the evolution of the colouring of bleached palm oil depending on the factors.

Table 15. Validation of the decolouration model.

Validation elements	Obtained values	Standard values	Acceptable values
R^2	0.95	1	≥ 0.90
ajusted R^2	0.90	1	≥0.80
AADM	0.00789217	0	0 – 0.3
Bf	1.0000546	1	0.75 – 1.25
Af	1.0000546	1	0.75 – 1.25

3.2. Optimization of Decolouration

3.2.1. Influence of Temperature Levels and Quality of Decolourizing Earths on the Color of Bleached Oil

Figure 1 (a, b, c) represents the iso-response curves and the surface response curves of colour variation after bleaching with the use of Indian decolourizing earth with a raw material of DOBI 2.3. It is observed that this earth has the colour variation contour lines at the high level of 16.0 red and at the low level of 15.3 red. The optimum value obtained is 18.7 red. It is also observed that the values of the colour responses increase with the introduction temperature of phosphoric acid, the percentage of phosphoric acid and the introduction temperature of the decolourizing earth. This could be explained by the fact that high temperatures promote the decomposition of carotenoïds. Indeed, heat treatments can affect the integrity of carotenoïds [17]. In addition, the increase in temperature contributes in reducing the viscosity of the oil, thus esuring better dispersion of particles and the maximum fixation of gums likely to be found in palm oil [7, 15]. It also appears that the reduction in colour is influenced by the effect of opposite temperature levels: the low level of the first temperature and the high level of the second temperature are an illustration of this with a high colour around 16.2. Therefore, the minimum temperature for introducing phosphoric acid should not be used because it contributes to reducing its effectiveness. However, the temperature at which the earth is introduced should not exceed 105 ℃ because the higher temperatures during decolouration could cause colour reversion.

Furthermore, these values of the colour variation contour lines are higher than those obtained by [14] with American decolourizing earth with values of high level of 14.4 red and low level of 13 red. This shows that the increase in colour depends on the decolourizing earth used. This difference in values could be explained by the fact that American earth presents a better performance than Indian earth, with an optimum value of 15.6. This particularity given to American earth is due to its specific characterisation (high acidity, pH). This characteristic ensures better fixation of the colored compounds and gums found in crude palm oil. This fixation can be understood by the affinity of the anions (gums fixed by H_3PO_4) for the metal ions on the surface of the adsorbent such as Ca^{2+}, Mg^{2+} which contributes to better fixation of these compounds. Furthermore, [18] and [1] showed that the acid concentration is the most important factor in the bleaching process and that the improvement in the bleaching power of clay accompanies the increase in the concentration of the solution. Indeed, the acid activation of the decolourizing earth increases its specific exchange surface and its porosity [7]. This contributes to effective absorption of colored compounds and gums.

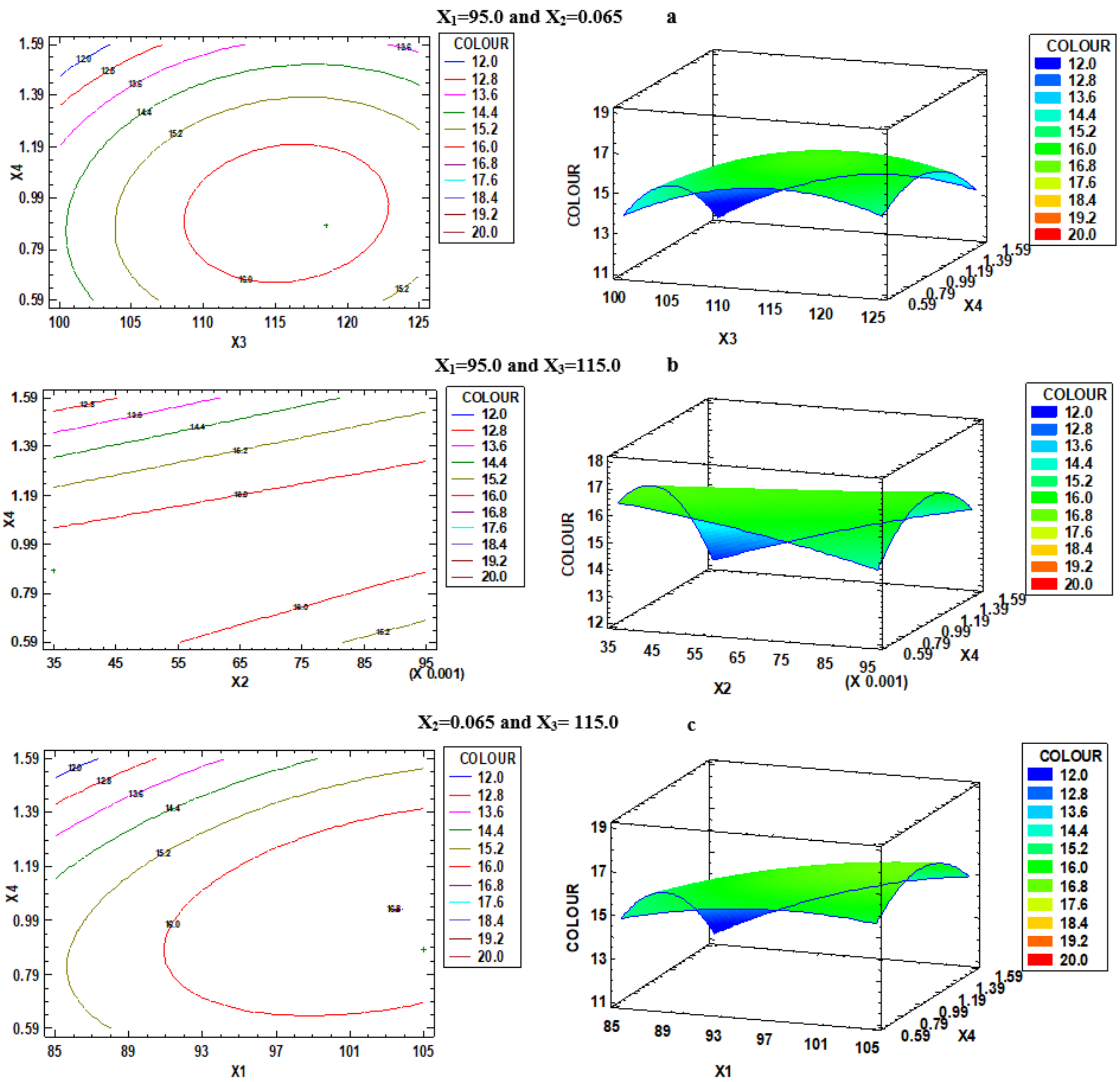

Figure 1*. Iso-response and surface response 3-D curves of colour variation with Indian earth and a DOBI 2.3 raw material.*

X_1: Introduction temperature of phosphoric acid (°C); X_2: Percentage of phosphoric acid (%); X_3: Introduction temperature of the decolourizing earth (°C); X_4: Percentage of decolourizing earth (%)

3.2.2. Influence of Temperature Levels and Quality of Palm Oil on the Colour Bleached Oil

Figure 2 (a, b, c, d, e, f) and figure 3 (a, b, c, d, e, f) show, respectively, the contour curves and the surface response 3-D curves of the red colour obtained after bleaching, with American earth and the use of the same raw material of DOBI 1.3, but at high and low temperature levels. We note that the bleaching temperature, when at its high level (120 ℃), gives a color of 15.8 red. At its low level (110 ℃), it gives a colour of 17.6 red. These values are also higher than those obtained by [14] with American earth and DOBI 2.3 oil, with high level values of 14.2 red and low level of 15.4 red.

These contour lines graphically show that temperature influences the colour of the bleached oil depending on the raw material. This could be explained by the fact that the bleaching temperature depends on the quality of the raw material. The degradation of the raw material involves the existence of oxidation compounds, which, in the presence of high temperatures, cause dark decolourations. These oxidation compounds are difficult to extract during refining. In addition, during refining, they polymerise under the action of high temperatures and make the refined oil dark [5, 3]. DOBI is a very important parameter for predicting the refining suitability of crude palm oil. Crude palm oil with a DOBI value (≤1,8) is difficult to refine due to the presence of oxidation compounds which are difficult to remove during refining [10]. This can lead to instability of the colour of palm olein obtained from degraded palm oils. It is therfore necessary to know the value of DOBI before refining.

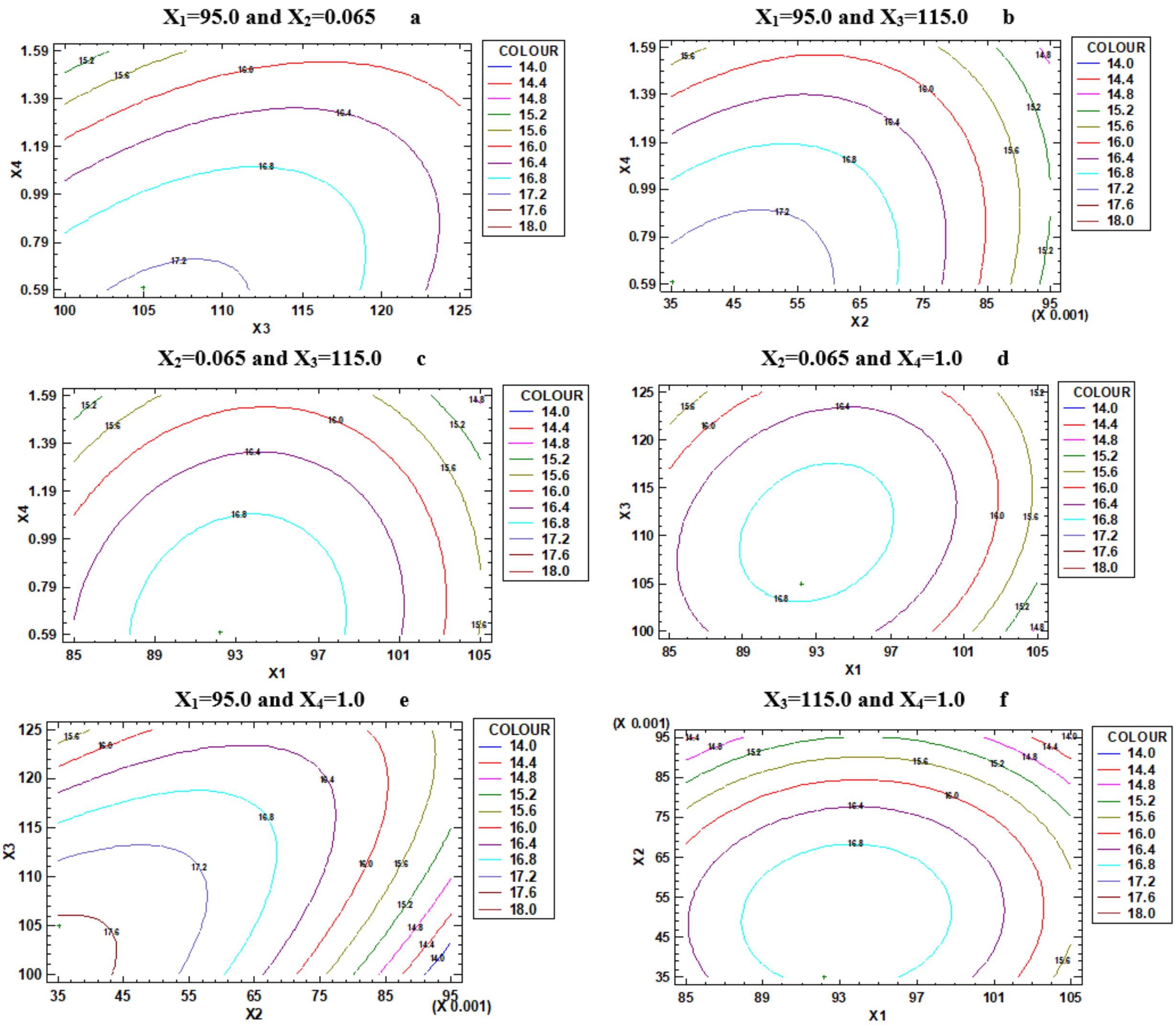

Figure 2. Iso-response curves of colour variation with American earth and a DOBI 1.3 raw material.

X_1: Introduction temperature of phosphoric acid (°C); X_2: Percentage of phosphoric acid (%); X_3: Introduction temperature of the decolourizing earth (°C); X_4: Percentage of decolourizing earth (%)

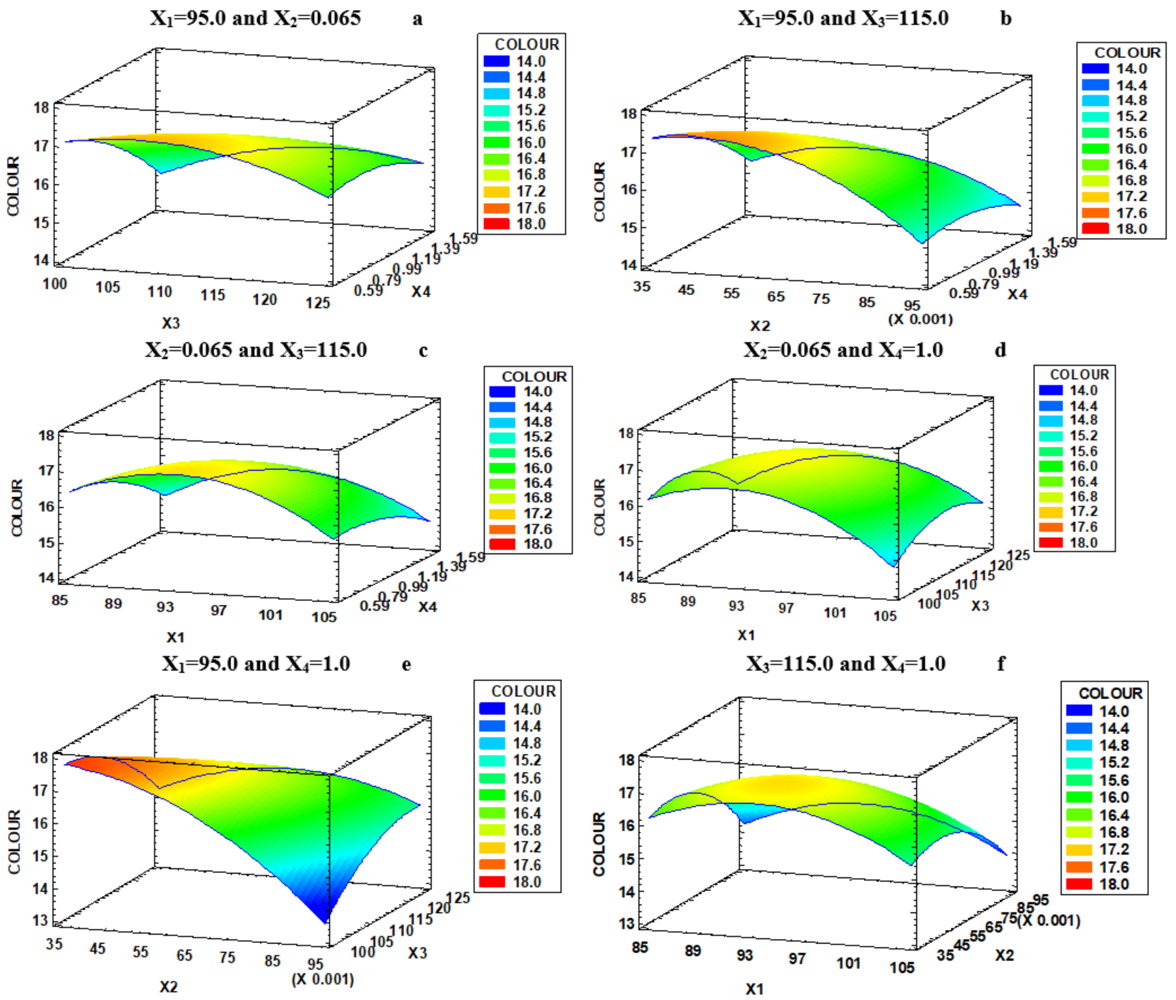

Figure 3. *Surface response 3-D curves of colour variation with American earth and a DOBI 1.3 raw material.*

X_1: Introduction temperature of phosphoric acid (°C); X_2: Percentage of phosphoric acid (%); X_3: Introduction temperature of the decolourizing earth (°C); X_4: Percentage of decolourizing earth (%)

4. Conclusion

The objective of this work was to optimize the combined use of raw material, decolourizing earth and temperature on the colour of bleached palm oil. Two different decolourizing earths (American and Indian) were used, as well as two different DOBI palm oils (2.3 and 1.3). The influence of these three parameters on the colour of bleached palm oil was evaluated using four-factor composite experimental designs. The results obtained indicate that the Indian earth with palm oil of DOBI 2.3 has the contour lines of colour variation at the high level of 16.0 red and at the low level of 15.3 red. Colour response values increase with temperature and percentage of phosphoric acid. The decrease in colour around 15.4 is influenced by the effect of opposite temperature levels. The increase in colour depends on the decolourizing earth used. Indian earth binds less colored compounds and gums from crude palm oil. It remains less efficient than American earth.

The temperature influences the colour of the bleached oil depending on the raw material. The bleaching temperature, when at its high and low levels, result in a colour of 15.8 red and 17.6 red, respectively. Crude palm oil with a DOBI value of 1.3 is delicate to refine due to the presence of oxidation products.

The second-order polynomial models, with satisfactory validation in terms of R^2, adjusted R^2, AAMD, Bf and Af were generated and described the decolourization process. The optimal decolouration conditions (≤ 20 red max) of crude palm oil ($P \leq 0,05$) are: for american earth (with a DOBI 1.3 oil) 92.2 ℃ and 0.035% for temperature and percentage of phosphoric acid; 105 ℃ and 0.6% for temperature and percentage of decolourizing earth. For Indian earth (with a DOBI 2.3 oil) 105 ℃ and 0.035% for temperature and percentage of phosphoric acid; 118.5 ℃ and 0.88% for temperature and percentage of decolourizing earth. These optima are recommended for the decolourization of oils of such quality.

Abbreviations

CPO	Crude Palm Oil
BPO	Bleached Palm Oil
DOBI	Deterioration of Bleachability Index
DOBI 1.3	Deterioration of Bleachability Index 1.3
DOBI 2.3	Deterioration of Bleachability Index 2.3
RBD	Refined Bleached Deodorized Oil
YAde	American Decolourizing Earth Response
YIde	Indian Decolourizing Earth Response
R^2	Correlation Coefficient
AAMD	Absolute Mean Deviation Analysis
Bf	Bias Factor
Af	Accuracy Factor

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Author Contributions

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Crépin Ella Missang: Supervision, Validation, Visualization

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Conflicts of Interest

The authors declare no conflicts of interest.

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