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ORIGINAL PAPER

Optimization of fat bleaching in soap production: from laboratory to industrial scale

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Abstract
This paper reports the use of design of experiments (DOE) with the objective of finding optimal industrial conditions for fat bleaching in soaps production. Laboratory experiments were performed evaluating the effects of residence time, temperature and pressure on the resulting fat color by two different colors scales: red (R) and yellow (Y), through light emitted or reflected by the mixture. Statistical analysis as well as a visual analysis of response surfaces indicated that only factors "Time", "Temperature", "Pressure" and "Time × Temperature" presented considerable effects on the total color, and an operation with long residence time, low pressures and low temperatures would be ideal for removal of the R and Y colors in an easy and effective manner. The responses found in these experiments were reproduced in industrial equipment, and the fat color results obtained were within the indicated experimental value. This approach is of simple laboratory execution, good reproducibility at the industrial level and also with good fat bleaching efficiency. Our finds indicated that 50 min of residence time, 70 °C and 6.7 kPa would be ideal operation conditions in industrial scale.

Keywords Fat bleaching · Refining · Animal fat

Introduction

Soap is one of the oldest personal care products used by man, since there is evidence of its production through animal fat and ashes from about 2800 BC and its use as cleaning material for at least 1000 years (Poucher 1993). Although it has accompanied many changes in mankind, soap formulation has hardly changed, as it is a simple reaction, with reagents easy to find: neutral fats (esters) and alkalis (most sodium or potassium). One of the most expensive steps in the soap production process is the fat bleaching, since for the most part, animal sebum purchased from large industries (representing more than 90% of the educts) is not found in acceptable color, appearance and odor conditions to derive a consumer good for personal hygiene, which leads to a need for cleaning and reducing strong odors of the raw material (Spitz 2016; van Duijn 2016).

Fat bleaching is an adsorption of the polar impurities (bone, blood, free acids) found in nonpolar fat (Chew et al. 2017; Taylor 2005). Clays are used as a cheaper option of fat refining in the soap production. They are hydrophilic, but may contain hydrophobic sites if there are insertions of ions that alter this characteristic (Kuuluvainen et al. 2015a). In Brazil, the most frequent mineral for this procedure is bentonite clay (Asgari et al. 2018; Olu-Owolabi et al. 2016), which originates what is industrially popularly known as "bleaching earth" (Kuuluvainen et al. 2015b), used in this study.

The most important variables in fat bleaching are residence time, temperature and pressure. Some researches states 20–30 min of residence time would be ideal for satisfactory results (Kaynak et al. 2004), whereas other claim that 50 min would be the best residence time (García-Moreno et al. 2013). Some authors suggest the ideal temperature for adsorption would be between 90 and 115 °C (Aachary et al. 2016; Kaynak et al. 2004), while others suggest that temperature should be lower, turning around 70 °C (Igansi 2018). Thus, there is no consensus on the exact values of temperature and residence time for the fat bleaching, but only a targeting for approximate values.

Brazil is the third largest consumer of cosmetics in the world and the second largest soap bar consumer. However, despite the high consumption compared to other countries, the soap market can still be very exploited, as there are

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several opportunities for growth within Brazil. Currently, there are basically two types of products present in the Brazilian market: "pure vegetable" soaps, which do not use animal fat in their formula, have higher added value and, therefore, are viewed from a presentable and luxurious perspective, and soaps from animal fat, which use sebum as a raw material, have a lower cost and are easily found in large retail stores.

It is known that "bleaching earth" has a very high cost and its use in industries is usually not optimized, since little research was done within the subject to find its best operating parameters. Therefore, it is necessary for soap industries to do an analysis and optimization of its fat bleaching process. The objective of this work is to evaluate the effect of temperature, residence time and pressure on the bleaching of bovine fat, in order to provide directions for the best industrial operation. This paper used the operational conditions found by the DOE as guidelines for industrial-level research, in order to prove the effectiveness of the proposed analysis. Our research provides a simple and efficient step-by-step for industrial fat bleaching.

Methodology

Materials and color measurements

In this study fat (sebum) from cattle slaughterhouse in the region of Campinas, São Paulo, Brazil, was used. The adsorbent was the bentonite clay, brand Tonsil, from Clariant. To perform the color analysis, the Lovibond colorimeter (PFXi 995) was used, through two different colors scales: red (R) and yellow (Y), through light emitted or reflected by the mixture, which may have a value from 0 to 70. Hues can range from orange to browns, and in fats where yellow hues have a significant contribution to overall color along with the red component, the color index (Eq. 1) usually is calculated. Equation 1 was used to obtain the color index in each experiment:

$$\text{Color} = \frac{\frac{\text{ColorY}}{10} + \text{ColorR}}{2}. \quad (1)$$

Design of experiments (DOE)

There are three factors that are essential for a good fat bleaching: contact time between adsorbent and fluid, temperature and adsorption pressure (Chew et al. 2017; Egbuna et al. 2015). Therefore, these variables were chosen to ascertain the impact on the final fat color and then choose their best values. Thus, a 2^3 factorial design with duplicates was set up to understand both the impacts of the factors, but also their linearity, which represents the curvature of the surface. The DOE was performed in the laboratory and then the best data set was taken to the industrial plant.

For the variable "residence time", currently about 90 min are used in industrial processes. It has already been verified that, when this time is increased, the results of fat quality tend to worsen and, moreover some works recommended lower values (Kaynak et al. 2004). Residence time of 30 and 50 min were used based on different works. 70 °C and 115 °C were chosen for the levels of temperature. The pressure analysis was carried out qualitatively (categorical variable), with high and low pressure, through filtration at ambient pressure and vacuum. Table 1 provides the full factorial design used in this work.

Laboratory and industrial-scale experiments

For each 100 g sample of crude fat, 3 g of clay was placed. The mixture was then heated for 10 min with stirring. From this moment to the rest of the time was used in the oven at the chosen temperature. After that the mixture was filtered by room pressure in some experiments and vacuum in the others. The bleached fat sample was then placed in a cuvette for color measurement in the colorimeter. All of the above procedure was repeated for each condition chosen.

After determining the best factors for laboratory experiments, the results were tested in industrial scale. The equipment used was a cylindrical agitated tank of approximately 29 m^3 volume, 1.2 m of radius and 6.5 m height. Its agitator has 15 Hp of power, and works at 1755 rpm. Filtration occurs by means of a filter press of approximately 3 m in height by 6 m in length. In this case, the best time and temperature values were tested at high or low pressure variables, 16 kPa or 6.7 kPa, respectively, depending on the DOE result. These results were compared with the experimental ones to verify reproducibility at the industrial level.

Table 1 Design of experiments according to the chosen variables

Experiment	Time (A) (°C)	Temperature (B) (°C)	Pressure (C)
1	30	70	High
2	40	92.5	Low
3	50	70	Low
4	50	115	High
5	30	115	High
6	30	70	Low
7	50	70	High
8	40	92.5	High
9	30	115	Low
10	50	115	Low

Results and discussion

The results obtained from the experiments can be found in Table 2. In order to reconcile the effects of the factors in both yellow and red colors, the color indexes were calculated by Eq. 1 and represented in the last column of Table 2. The data described in Table 3 were obtained from the Minitab analysis of variance (ANOVA), used in this work. It is known that only the factors with p value less than $\alpha = 0.05$ are relevant for the analysis, i.e., in this case, the factors "Time", "Temperature", "Pressure" and "Time × Temperature".

The fact that the other interactions have higher p value means that either there is no physical meaning for such combinations or that they do not significantly change the color result of the sample. In addition, it is also verified that the curvature, result of the analysis of the central point, is expressive for the understanding of the behavior as a whole, which will be done with the help of its coefficient. The understanding of the best parameters then occurs through the coded coefficients, also provided by the software (Table 4).

From Table 4, it is possible to note that for the time its coefficient is negative, this represents that the larger the time variable, the smaller the color obtained at the end. It is also the largest module coefficient, representing that it has the greatest influence on the final result. This is mainly due to the adsorption itself, which takes some time at each stage and, with shorter times, the process should not be concluded. For the temperature, which presented a positive coefficient, it is verified that the higher temperature level favors the higher color obtained, showing that the interest is lower values for this variable. It is worth considering that a very wide temperature range has been used, so there may be other suitable intermediate values. What has been observed is that the higher level favors a darkening of the sample, most likely by the oxidation reactions that are susceptible to occur. It is also important to highlight that the clay itself is composed of different highly activated oxides (Chew et al. 2017), so it can act against bleaching itself if it is arranged under unfavorable conditions. However, the increase in temperature also causes a reduction in the fat viscosity, facilitating contact

Table 2 Average color results obtained by DOE

Run	Time (A)	Temperature (B)	Pressure (C)	Color Y	Color R	Color
1	30	70	High	24	1.31	1.86
2	40	92.5	Low	23.5	1.14	1.75
3	50	70	Low	11.5	0.92	1.04
4	50	115	High	17	1.07	1.39
5	30	115	High	33.5	2.32	2.84
6	30	70	Low	18	1.1	1.45
7	50	70	High	15	1.2	1.35
8	40	92.5	High	32.5	1.25	2.25
9	30	115	Low	28	1.6	2.20
10	50	115	Low	13	1.0	1.15

Table 3 DOE analysis of variance (ANOVA)

Source	GL	SQ (Aj.)	QM (Aj.)	*F*	*P*
Model	8	2.911	0.365	646.761	0.030
Linear	3	2.403	0.801	1424.670	0.019
Time	1	1.487	1.487	2645.000	0.012
Temperature	1	0.475	0.475	845.000	0.022
Pressure	1	0.441	0.441	784.000	0.023
Interactions by 2 factor	3	0.327	0.108	194.630	0.053
Time × Temperature	1	0.301	0.300	533.890	0.028
Time × Pressure	1	0.025	0.025	45.000	0.094
Temperature × Pressure	1	0.003	0.003	5.000	0.269
Interactions by 3 factors	1	0.015	0.015	27.220	0.121
Time × Temperature × Pressure	1	0.015	0.015	27.221	0.121
Curvature	1	0.162	0.163	289.000	0.037
Error	1	0.001	0.001		
Total	9	2.912			

Table 4 DOE-coded coefficients provided by Minitab

Term	Effect	Coefficient	EP of coefficient	T value	P value	VIF
Constant		1.656	0.0084	197.5	0.003	
Time	− 0.864	− 0.443	0.0084	− 51.4	0.012	1
Temperature	0.487	0.243	0.0084	29.1	0.022	1
Pressure	0.420	0.210	0.0075	28.0	0.023	1
Time × Temperature	− 0.388	− 0.194	0.0084	− 23.1	0.028	1
Time × Pressure	− 0.113	− 0.056	0.0084	− 6.7	0.094	1
Temperature × Pressure	0.038	0.019	0.0084	2.2	0.268	1
Time × Temperature × Pressure	− 0.088	− 0.044	0.0084	− 5.2	0.121	1
Central point		0.319	0.0188	17.0	0.037	1

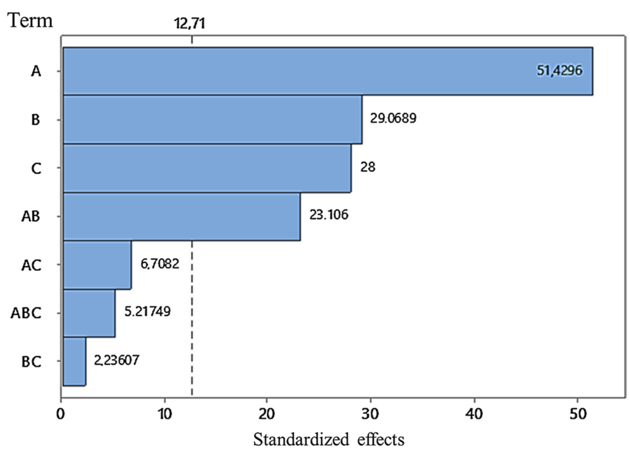

Fig. 1 Pareto diagram of the effects expressed in module and standardized obtained from Minitab

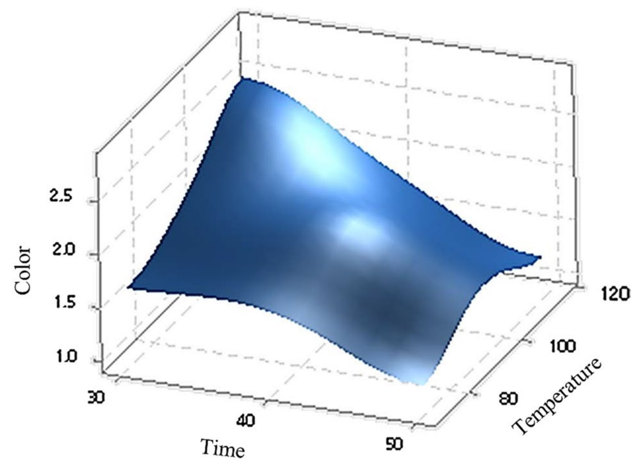

Fig. 2 Surface chart of influence of time and temperature on color

with the adsorbent, so a diagnosis of these considerations should be made.

Regarding pressure, since this factor was qualitative, there were only two levels (high and low). Its positive coefficient shows that high pressures increase the fat color, as opposed to what is of interest. The lower concentration (lower pressure) of air in the bleach prevents the oxidation and hydrolysis of fats, becoming more favorable (Larouci et al. 2015). Analyzing the interaction "Time × Temperature" it is verified that it has negative coefficient, showing that the combination between time and temperature can reduce the final coloration. Figure 1 illustrates the effect of each factor evaluated; where "A" represents the residence time effect, "B" the temperature effect and "C" the pressure effect. Those that have value above the red and dashed lines are treated as relevant, which are quoted above. However, a better understanding of the effect of each factor can be given by the surface chart between time, temperature and color, bringing a three-dimensional view of the problem by the most influential factors.

Through Fig. 2 it is possible to identify that the best factors for obtaining clear bleached fat are "higher time" and "lower temperature", in this case, represented by 50 min and 70 °C. In addition, it is seen that the change in time leads to much more drastic changes than the change in temperature, proving the hypothesis of the coefficients that the time effect was of greater magnitude than the effect of temperature. Other important information that can be abstracted from this figure is that, despite three-dimensionality, it has only slight concave and convex indicia, a result of the center point coefficient. This shows that although a *p* value less than *alpha*, one variable should have a greater influence on the curvature than the others, and this would be the temperature (where the center point is farthest from the extremity line), according to Fig. 3.

For the variables "time" and "temperature", contour plots for the red, yellow, and the general color are described in Figs. 4, 5 and 6, respectively. Analyzing Figs. 4 and 5, it can be seen that for each color, R and Y, time exerts a great influence, since there is a clear distinction of color as it evolves. Higher the residence times, the higher the reduction of its colors. Temperature seems very important factor too, especially in combination with time, which can either be extremely favorable or very harmful. Figure 4 shows that,

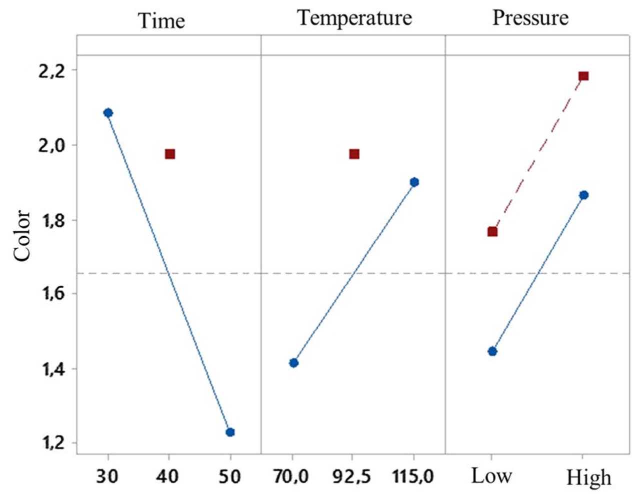

Fig. 3 Effects of the center point associated with the end points

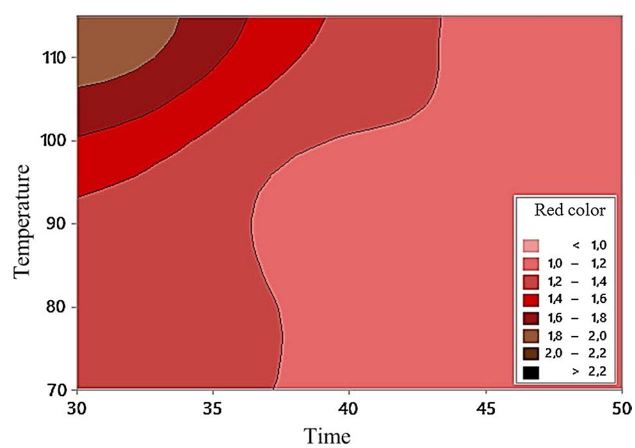

Fig. 4 Contour diagram of red color as a function of processing time and temperature

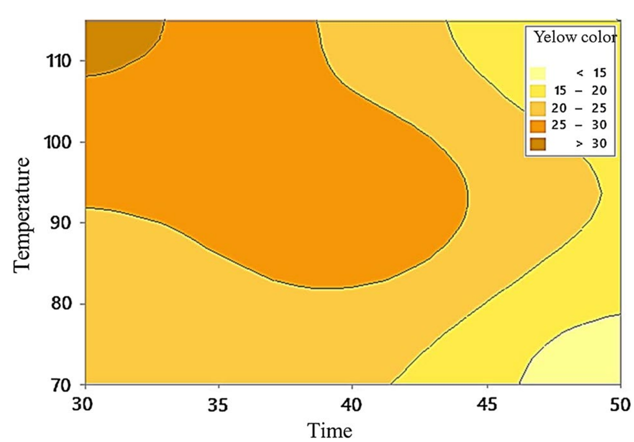

Fig. 5 Diagram of yellow color contour as a function of the processing time and temperature

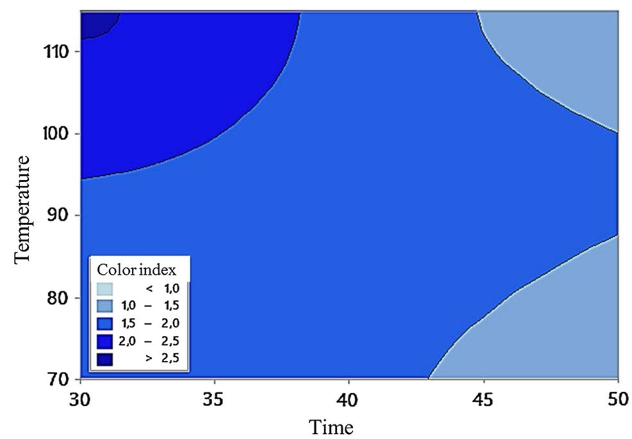

Fig. 6 General color contour as a function of processing time and temperature

from about 43 min, there is no variation of the red color for all temperatures analyzed, being the lowest red color obtained during the bleaching. This may be because the reddish sebum residues are removed for a certain period of the adsorption and the remainders will not be removed regardless of the time, either because of size, polarity, or other aspects that may disrupt the process (Asgari et al. 2018; Chew et al. 2017; Larouci et al. 2015). However, for the yellow color, it can be seen in Fig. 5 that the most reduction was achieved at the lowest temperature, suggesting that is better to operate at such condition.

Figure 6 summarizes the behavior of the two colors using the index calculated by Eq. 1. For better quality of the bleached fat, the residence time should be around 50 min, because it provides the lowest color at both limits (high and low temperatures). However, temperature must be around such limits to achieve the lowest general color. Due to the results of reduction of yellow color described in Fig. 5, for the best reduction possible, temperature should be lower, around 70 °C, favoring the fat bleaching in both Y and R. It is important to highlight that this result is very important considering energy cost reduction. Although it was not possible to insert the variable pressure because it was categorical in the experiments, the coefficient obtained indicates that lower pressure favors the color reduction, which helps to avoid the contact of fats with water and oxygen, completing the analysis more trivial that must be made of this DOE. Equation 2 provides the DOE adjustment of total color reduction as a function of the factors analyzed.

$$\text{Color} = 1.656 - 0.433x_1 + 0.243x_2 + 0.210x_3 - 0.194x_1x_2 - 0.05x_1x_3 + 0.019x_2x_3 - 0.044x_1x_2x_3, \quad (2)$$

where x_1, x_2 and x_3 represent the residence time, temperature and pressure effect, respectively.

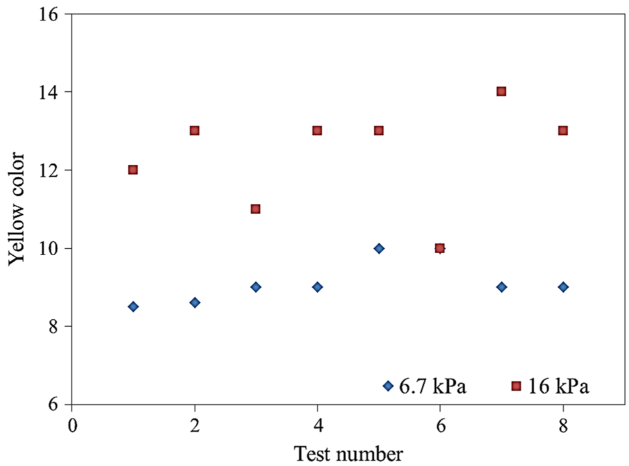

Fig. 7 Comparison of yellow color for different pressure values

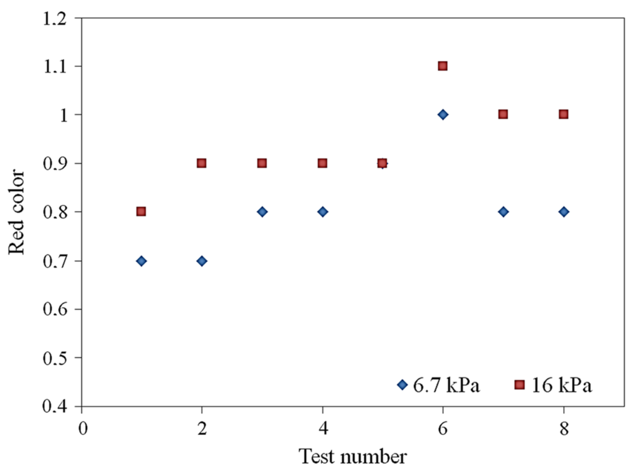

Fig. 8 Red color comparison for different pressure values

There is no way to perform an industrial process optimization without testing the assumptions in the plant itself. Therefore, after the DOE, eight tests were made by the conditions that were considered optimal in an industrial plant (temperature of 70 °C and residence time equals 50 min), to validate them and to define the pressure point. As it was found that the lower the pressure the greater the bleaching efficiency, it was tested two values of the variable: 16 kPa (value commonly used in industrial operations) and 6.7 kPa (minimum reached by the equipment used in this work). The results of yellow and red color are described in Figs. 7 and 8, and confirmed what were obtained in the experimental DOE. For all tests performed in industrial plant, the lower pressure provided the lower color Y and R. The results obtained in this study are lower than the values normally accepted for both evaluated colors. Desirable values for red and yellow colors are 3 and 10, respectively (Foletto et al. 2011; García-Moreno et al. 2013; Riyadi et al. 2016; Silva et al. 2014). Based on the results shown in these figures, Table 5 summarizes the colors obtained using the best operational conditions in experimental and industrial plant.

Conclusions

The main objective of this work was to identify the influence of residence time, temperature and pressure on the fat bleaching process by design of experiments (DOE). It was considered a residence time similar to those used in industrial processes, verifying that the longer residence time provides greater fat bleaching. Temperature effect results indicated better operations with low values. Finally, low pressure operation was indicated in order to provide low values of fat color. This paper also reported the inclusion of operational conditions found from the DOE in an industrial bleaching plant, in order to evaluate the effectiveness of the proposed analysis. It was found that residence time of 50 min, temperature of 70 °C and pressure of 6.7 kPa provided the most pronounced fat color reduction, following what was expected by experimental results, although the pressure effect analysis was performed using categorical variables.

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Table 5 Comparison of the color achieved using the optimum conditions of fat bleaching in experimental and industrial tests

Experimental red color	Industrial red color	Error (%)	Experimental yellow color	Industrial yellow color	Error (%)
7.954 ± 0.765	9.125 ± 0.582	14.72	0.768 ± 0.18	0.813 ± 0.1	5.79

Time = 50 min, temperature = 70 °C and pressure = 6.7 kPa

Compliance with ethical standards

Conflict of interest All authors have participated in (a) conception and design, or analysis and interpretation of the data; (b) drafting the article or revising it critically for important intellectual content; and (c) approval of the final version.

Authorship conformation form This manuscript has not been submitted to, nor is under review at, another journal or other publishing venue.

The authors have no affiliation with any organization with a direct or indirect financial interest in the subject matter discussed in the manuscript.

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