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Preliminary study regarding the optimisation of the accelerated ageing of sugar cane spirit by applying ultrasound-assisted extraction and white oak chips (Quercus Alba)

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Abstract

The ageing of alcoholic beverages is a common practice in order to increase their quality due to the release of wood extractable compounds from the barrels used. However, this process is slow and can take several years. This research focussed on optimising the use of *Quercus alba* chips and ultrasound in the accelerated ageing process of sugar cane spirit using the response surface methodology. The effect of temperature and ultrasound was analysed using a two-level factorial design type 2^2. Subsequently, the accelerated ageing process was optimised using response surface methodology. Temperature and time of exposure to ultrasound energy were the selected independent variables. As a response variable, the total polyphenol index (TPI) was selected. In addition, the CIELab method was used to determine the colour characteristics (L*, a*, b*) of the aged samples. Roasting had a negative effect on the TPI, while the oak chips that were untoasted and treated with ultrasound obtained the highest TPI yield (80.5–81.5 mg GAEeq/L). The TPI values obtained through the use of the accelerated ageing method suggest that it is more efficient than the traditional ageing process, as it leads to a decrease in the maturation period of sugar cane spirit. Regarding the colour properties, a higher intensity of colour was found in the samples where the accelerated ageing method was applied. The temperature and sonication time of 48.9°C and 18.8 min were the optimal parameters to obtain a TPI value (85 GAE_{eq}./L) comparable to the Brandy Solera Reserve (12–18 months for the traditional method) for sugar cane spirit using the accelerated method. The findings of this research can contribute towards the development of new ageing processes of distillates, mainly in terms of decreasing ageing times.

1. Introduction

Ageing distilled spirits is a common practice that has been used for centuries in order to improve a beverage's flavour, such as rum, whisky, brandy and wine spirits (Silvello & Alcarde, 2020). Each beverage has a characteristic aromatic profile that is influenced by various factors including raw material, fermentation process, distillation operation and ageing or maturation. In this last stage, the distillate improves the flavour due to the extraction of aromatic molecules from the wood barrels. The phenolic compounds (phenolic acids and aldehydes) are mainly composed of vanillin, syringaldehyde, coniferaldehyde, gallic acid and vanillic acid. In general, the samples subjected to longer ageing times present the highest concentrations of these compounds (Bortoletto & Alcarde, 2013; Canas, 2017). All the same, the relationship between ageing time and phenolic compound content is not a simple one (Wiśniewska et al., 2015). The content of these biomolecules in the final product is affected by several factors, like the wood's region of origin, age and species as well as the heat treatment applied to the wooden barrel production. Regarding the type of wood, species belonging to the genus *Quercus* (*Q. alba, Q. robur, Q. petraea*), commonly known as oak, are traditionally used for this purpose. Nonetheless, the selection of species is often influenced by the region where the beverage is processed. For example, in North America, *Q. alba, Q. garryana, Q. macrocarpa* and *Q. stellate* are used, in Colombia *Q. humboldtii* is utilised and in Europe, others such as *Q. petraea, Q. robur, Q. cerris, Q. suber, Q. lyrata, Q. bicolor* and *Q. lanuginose* are applied for the cooperage.

However, traditional ageing involves long periods of time and large storage areas, which leads to an increase in the price of the final product (Jordão & Cosme, 2022). In recent years, several ageing alternatives have been studied to obtain results from the point of view of organoleptic properties while reducing ageing time. A very simple but effective method is the application of oak fragments, such as chips, cubes, staves, blocks or powders. Several authors compared this alternative with the traditional barrel ageing and obtained similar organoleptic results in less contact time (Costa et al., 2020; de Coninck et al., 2006; Laqui-Estaña et al., 2019). Other researchers chose to accelerate the ageing process using micro-oxygenation, consisting of the controlled introduction of a small oxygen dosage. They reported positive results on the wine's colour and phenolic evolution, compared to wine aged using the traditional method, when the treatment was applied correctly (Martínez-Gil et al., 2020; Oberholster et al., 2015; Sánchez-Gómez et al., 2018). It is worth mentioning that these studies focused on red wine and that sugar cane spirit has scarcely been studied until now.

In addition, the application of ultrasound energy has been used to accelerate the ageing process, as in the studies of Schwarz et al. (2014) and Delgado-González et al. (2017), who applied it to the ageing of brandy. These authors concluded that using a combination of ultrasound waves and oak chips, it is possible to produce brandy with similar characteristics to that which was aged using a traditional process but with significant savings as regards time.

This work aimed to optimise the use of *Q. alba* chips and ultrasound in the accelerated ageing process of sugar cane spirit using the response surface methodology. Previously, we applied a Two-Level Factorial Design (TLFD) to estimate the effects of chip toasting and ultrasound energy in the phenolic extraction from *Q. alba* wood.

2. Materials And Methods

2.1. Sugar cane spirit and preparation of Q. alba chips.

We purchased the sugar cane spirit from an artisanal producer in Puyo, Pastaza Province, in the Ecuadorian Amazon region. The sample was obtained using a column of simple distillation with an alcohol content of ~ 54% v/v (ethanol/water). Meanwhile, the *Q. alba* wood was supplied by the *Trovador* distillery, Tungurahua, Ecuador, and cut into chips of 100x100x100 mm. Two types of chips were used in the ageing experiments: untoasted and toasted. The toasted chips were subjected to a temperature of 180°C for 3 h according to Monedero et al. (1998) inside a stove (Model: SFE 700, Memmert, Schwabach, Germany).

2.2. Ageing methods for sugarcane spirit

Two laboratory-scale methods for ageing sugar cane spirit were used: the static method and the dynamic method accelerated by ultrasound-assisted extraction (UAE). The static method consisted of 1 L (sugarcane spirit) in amber bottles, to which 12 g of toasted or untoasted *Q. alba* chips were added and this was left standing for 30 days. Samples for analysis were taken every 7 days. Meanwhile, the

dynamic method accelerated by UAE consisted of a Digital Ultrasonic Cleaner-set (400 W/L and 40 kHz, model: WUC-D06H, Daihan Scientific Co., LTD, Seoul, South Korea), a glass cylinder (containing 12 g of *Q. alba* chips) and a tank (5 L) for storing. In this system, 1 L of sugarcane spirit was recirculated by means of a submersible pump at a flow rate of 50 L/h. The UAE experiment had a total duration of 7 h, divided into cycles of 15 min exposition to ultrasound and 45 min without ultrasound, keeping the sugarcane spirit recirculating all the while. Figure 1 shows the scheme that the system used.

2.3. Total Polyphenol Index (TPI)

TPI was determined using the Folin-Ciocalteu Method according to that which was outlined by Singleton et al. (1999), with slight modifications. In short, 10 mL of the sample was diluted in 25 mL methanol (70:30 v/v), which will henceforth be referred to as stock solution. Then, 150 µL of stock solution, 2400 µL of distillated water and 150 µL of the Folin-Ciocalteu reagent (0.25 N) were added to a volumetric flask. The mixture was stirred for 3 min and allowed to react for 5 min. Next, 300 µL of Na_2CO_3 solution was added and it was incubated again for 2 h at room temperature in the dark. The blank was prepared in the same manner but without adding the sample. The absorbance was measured at a wavelength of 765 nm in a Thermo Spectronic Genesys 5 UV-Vis Spectrophotometer (Thermo Fisher Scientific Inc. Waltham, MA, USA). A Gallic acid calibration curve (10–150 mg/L) was created and results were expressed in mg equivalent of Gallic acid (mg GAE_{eq}./L). All the measurements were performed in triplicate. All chemicals and solvents were of analytical grade and were purchased from Sigma-Aldrich Co. (St. Louis, MO, USA).

2.4. Colour parameter determination

The colour parameter of the aged spirit samples was analysed by visible spectrometry. The absorbance of the aged samples was measured using a Spectronic Brand UV-Visible spectrophotometer, model Genesys 5, at wavelengths of 450, 520, 570 and 630 nm. The CIELab Method was used to calculate the colourimetric coordinates (Glories, 1984). The MSCV software (available in: https://www.unirioja.es/color/descargas.shtml), developed by the Research Colour Group at the University of La Rioja for the colour of wine, was used to calculate the colour CIELab coordinates. These parameters are L* (clarity), a* (from green to red), b* (from blue to yellow) and C* (chroma or saturation). Finally, the approximate colour corresponding to the CIELab chromatic obtained for each sample was visualised as a reference.

2.4. Experimental design

Two design methods were used in the experimental planning: factor screening and process optimisation. In the first stage, a TLFD type 2^2 with three replicates was used as the exploratory mode. The advantage of this design type is the low quantity of experiments needed while still allowing one to obtain the estimation of the independent variable effects with the same accuracy as other more complex designs. The factorial design provides contrasts of averages, as well as the quantification of statistical power to the effect estimates. Also, it reveals the effect of the interactions of the factors, which often proves to be the key to understanding a process (Anderson & Whitcomb, 2017). The parameters, chip type (untoasted and toasted) and ageing method (the static method and the dynamic method faccelerated by UAE) were

selected as independent variables and TPI as a response variable. Analysis of variance (ANOVA), Pareto chart and Bonferroni limit were used to assess the influence of the simple effects and their interactions on TPI. The experimental run setting is presented in Table 1, which also includes the response variables (experimental and predicted values).

Table 1. TLFD type 2^2 setting of the independent variables and corresponding experimental and predicted values of TPI

Run*	Chip types (A)	Ageing method (B)	TPI (mg $GAE_{eq.}$/L) (C)	
			Experimental	Predicted
1	Toasted**	Static	59.1	58.2
2	Untoasted	UAE	81.3	80.6
3	Untoasted	Static	65.8	65.9
4	Toasted	UAE	54.5	53.8
5	Untoasted	UAE	80.5	80.6
6	Untoasted	Static	65.1	65.9
7	Untoasted	UAE	80.1	80.6
8	Toasted	UAE	53.9	53.8
9	Toasted	UAE	53.1	53.8
10	Toasted	Static	54.5	58.2
11	Toasted	Static	60.9	58.2
12	Untoasted	Static	66.7	65.9

* Non-randomised.

** Level: Untoasted (-1) and Toasted (1); Static (-1) and UAE (1).

In the second experimental stage, a response surface methodology (RSM) type central composite design (CCD) was used for the optimisation of the process parameters. RSM is a widely used mathematical and statistical technique in the optimisation and evaluation of extraction processes. The CCD model for the planning and interpretation of the experiments was used.

The sonication time and temperature were the independent variables selected to optimise, whilst TPI remained as a response variable. The ageing procedure in this optimisation stage was similar to the first stage but with ultrasound times (UAE-t) of 10, 15 and 20 min. For the rest of the time, only recirculation

took place until a 1-hour cycle had been completed; each experimental run had a total time of 7 h. At the end of this period, the sample was taken to be analysed. With regard to the temperature, 30, 45 and 60 °C were the low, medium and high levels, respectively. Table 2 shows the experimental run setting used for optimisation (RSM type CCD). In total, there were 19 experimental runs, including the 12 factorial, 4 axial and 3 central points.

Table 2. RSM–CCD experimental planning for the optimisation of the UAE accelerated ageing of cane spirit

Run*	T (°C)	UAE-t (min)	TPI (mg $GAE_{eq.}$/L)	
	A	B	C	
			Experimental	Predicted
1	50	20	86.7	86.3
2	30	15	76.7	78.1
3	50	10	79.9	79.4
4	40	15	79.7	78.3
5	30	20	79.4	80.5
6	30	10	73.5	74.0
7	30	20	81.7	80.5
8	40	20	80.9	80.8
9	40	15	77.5	78.4
10	30	10	75.2	74.0
11	50	20	85.4	86.3
12	50	15	84.8	83.7
13	50	10	78.9	79.4
14	30	20	81.1	80.5
15	30	10	74.3	74.0
16	40	15	78.3	78.4
17	50	20	86.1	86.3
18	50	10	79.0	79.4
19	40	10	73.8	74.2

* Non-randomised.

** Level temp: 30 (-1); 40 (0); and 50 (1). Level ST 10 (-1); 15 (0); and 20 (1).

The multiple regression analysis was applied to evaluate the relationship between dependent variables and independent variables in order to predict the optimal point. A second-grade quadratic equation (Eq. 1) was employed to obtain the empirical model.

$$\mathrm{TPI}(\mathrm{mgGAE}_{eq.}/\mathrm{L}) = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1} \sum_{j=i+1} \beta_{ij} x_i x_j \; (1)$$

Whereβ_0, β_i, $\beta_{ii}$and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively. The termsx_i and x_j are the factors selected affecting the response variable (TPI) and k is the number of factors.

The software Design Expert Version 12 (Stat-Ease Inc., Minneapolis, USA) was used for the planning of both designs (factorial and optimisation designs). Design Expert randomises the order of the experimental runs, which contributes to guaranteeing that the model meets certain statistical assumptions and reducing the effects of factors not included in the design (Abreu Naranjo et al., 2018). In addition, analysis of variance (ANOVA) was applied to quantify the linear, quadratic and interaction effects of the independent variables on the response variable (TPI). The adjusted and predicted coefficients of determination and coefficient of variation (CV) were statistical parameters used to assess the robustness and precision of the model. The significance level at 95% ($p < 0.05$) and 99% ($p < 0.01$) were selected.

3. Results And Discussion

3.1. Model fit and estimation of the effect of the factors on the TPI

In the preliminary experiments of this investigation, the effects of the A and B factors and their interaction (AB) with the TPI were estimated by means of a TLFD. Table 3shows the ANOVA results applied to the variables of the factorial design for estimating the effects and significance.

Table 3
Main parameters of the ANOVA of the factorial design for the factors ageing method and type of chips

Parameter	Sum of Squares	df	Mean Square	F-value	p-value
Model	1247.4	3	415.8	133.3	< 0.0001
A-Chips type	892.9	1	892.9	286.2	< 0.0001
B-Ageing method	81.4	1	81.4	26.1	0.0009
AB	273.1	1	273.1	87.6	< 0.0001
Pure Error	25.5	8	3.12		
Cor. Total	1272.33	11			

The F-value of the model being 133.3 implies that the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. In addition, the factorial model fitted well with the R^2 (0.98) and the Predicted-R^2 (0.96) is in good agreement with the Adjusted-R^2 (0.97) with a smaller difference of 0.2, according to what was suggested by Anderson and Whitcomb (2017). The CV allows one to measure the precision and reproducibility of the model; lower values (CV <5%) indicate high precision and reproducibility of the experimental data (Liu et al., 2013; Martínez-Patiño et al., 2019). In this study, a low CV of 2.73% was obtained. Regarding the significance of the variables studied, the results indicate that both independent variables (A and B: $p < 0.0001$) and their interaction (AB: $p < 0.05$), have a significant effect on TPI. The effect levels can also be observed graphically in the Pareto chart (Figure 2) to facilitate understanding.

The lengths of the vertical bars are proportional to the absolute magnitude of the estimated effects, while the red line (Bonferroni limit) represents the minimum value of statistically significant effects with respect to TPI (95% of the confidence interval). It can be observed that chip type (factor A) and its interaction (factor AB) were the most significant factors for the TPI, both having negative effects, whereas ageing method (factor B) also was significant but had a smaller, positive effect. The negative effect of roasting on the polyphenol content can be explained by the fact that the toasted chips were subjected to a temperature of 180 °C for 3 h. Previous studies reported a decrease in the content of phenolic compounds when wood samples are subjected to temperatures greater than 60 °C, as the thermolabile phenolic compounds begin to degrade (Abreu-Naranjo et al., 2018; Deng et al., 2015). A comparable trend was reported by Kanakaki et al. (2015) in their study on wine ageing that focused on the effect that the degree of toasting of oak chips has on polyphenol extraction. These authors concluded in their study, that even moderate toasting provoked a drastic decrease in polyphenol content extracted from oak chips.

As can be seen in Table 1, for the combination of untoasted chips with ultrasound treatment, the highest TPI yield (80.5–81.5 mg GAE_{eq}/L) was achieved. Meanwhile, when the toasted chips and static ageing method were applied, the TPI values (54.5–60.9 mg GAE_{eq}/L) were the lowest. Regarding the TPI values obtained by the static and UAE methods in this study, the values reported by Muñoz et al. (2020) for brandy samples can be taken as a reference (Jerez-type brandies (58.7 mg GAE_{eq}./L) and Solera Reserve (86.7 mg GAE_{eq}./L)). The TPI values are equivalent to an ageing time of 6 to 9 months and 12 to 18 months by the traditional method, as established by the technical parameters of the Junta de Andalucía for Solera and Solera Reserve brandies, respectively.

The higher TPI extraction yields obtained with the application of the ultrasonic irradiations may be at the breakdown of cell walls, which allows an increased penetration of solvent at the cell contents and bioavailability of the analyte (Sanwal et al., 2022; Silva et al., 2022). It is worth mentioning that in addition to the effect of ultrasound on the ageing process of sugar cane spirit, the recirculation of the liquid on the wood helps to increase the extraction of phenolic compounds. Due to the turbulence created by the return of the liquid to the storage tank, which performs the function of micro-oxygenation, air is injected into the sample (Sánchez Guillén, 2016). These results are comparable with those reported by Delgado-González et al. (2017) in their study about the ageing of wine spirits. They concluded that the movement of distillate and the application of ultrasound waves have a synergic effect that increases the extraction of the wood's compounds. Thereby, it is highly effective in accelerating the extraction of bioactive compounds from natural sources in a shorter period of time. In this regard, it is important to consider this for the optimisation of UAE conditions as it is of high importance for maximising the accelerated ageing of sugar cane spirit.

3.2. Colour parameters of the ageing of sugar cane spirit by means of wood chips and ultrasound

Colour is a highly important parameter in the perception of beverages, because it plays a fundamental role in the consumer's first impression and influences their decision to select the beverage (Canas, 2017). The CIELab coordinates (L*, a*, b*) and a rough visual display of the colour are shown in Table 4.

Table 4. The CIELab coordinates and the calculated colour of the aged sample of sugar cane spirit

Run	L*	a*	b*	Calculated colour
1	97.0	-0.90	12.1	
2	98.4	-0.70	7.28	
3	97.9	-0.99	9.86	
4	98.5	-0.83	7.33	
5	98.1	-0.88	8.58	
6	98.0	-0.86	7.54	
7	98.8	-0.69	6.09	
8	97.2	-0.80	11.2	
9	98.3	-0.77	8.05	
10	98.3	-0.88	7.80	
11	95.5	-0.91	16.6	
12	98.3	-0.67	7.08	
13	98.2	-0.83	8.52	
14	98.5	-0.69	6.94	
15	97.5	-0.81	9.73	
16	97.1	-0.89	11.4	
17	96.9	-0.84	10.70	
18	98.4	-0.79	7.55	
19	97.6	-0.77	9.20	

Concerning the colour properties, a positive trend between the TPI and the colour was established (see Tables 2 and 4). Coordinate L* represents clarity, which is directly related to the visual sensation of luminosity. In samples 1, 8 and 11 (20 min and 50°C), the lowest values of L* were obtained, which means a higher intensity of colour. In addition, the values obtained for a* and b* indicate green and yellow hues, respectively, in all samples in general. The fresh sugar cane spirit is colourless due to the absence of phenolic compounds; the acquired colour changes are due to the ageing process. This is in accordance with the results of other beverages (Porto wine, brandy and red wine) reported by Bakker et al. (1986), Canas et al. (2000) and Baiano et al. (2016), respectively. Colour is an organoleptic attribute that allows identifying the degree of maturation and ageing and is usually used as a quality criterion (Lea & Piggott, 2003). Therefore, the application of the ageing treatment produces a sugar cane spirit with a more intense color. It can also be used as an indicator of the changes that occur during processing (Lea & Piggott, 2003).

3.3. Modelling and fitting of the response surface optimisation of ageing of sugar cane spirit for UAE

For the sake of the fit of the model, an ANOVA was applied with an RSM type CCD to the accelerated ageing process of the sugar cane spirit. The results are shown in Table 5.

Table 5
Main parameters of the ANOVA for the RSM type CCD design applied to the accelerated ageing process of the sugar cane spirit

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	283.7	5	56.71	60.86	< 0.0001
A- T	109.3	1	109.2	117.2	< 0.0001
B- UAE-t	154.8	1	154.8	166.2	< 0.0001
AB	0.1132	1	0.1132	0.1215	0.7330
A^2	18.99	1	18.99	20.38	0.0006
B^2	2.06	1	2.06	2.21	0.1607
Residual	12.11	13	0.9318		
Lack of Fit	3.99	3	1.33	1.64	0.2424
Pure Error	8.12	10	0.8122		
Cor. Total	295.7	18			

The F-value and p-value of the model (60.86 and < 0.0001) implies that the fit of the model is highly significant, demonstrating the relationship between the independent variables selected and TPI. Furthermore, the lack of fit of the model was not significant ($p < 0.05$), indicating that the model is adequate for representing the data inside the experimental space selected. P-values of less than 0.05 indicate model terms are significant. In this case, A, B and A^2 are significant model terms, whereas greater values ($p > 0.05$) indicate the model terms are not significant (AB and B^2). The values of the determination coefficients (R^2, predicted R^2 and adjusted R^2) were greater than 0.90, specifically 0.959, 0.910 and 0.943, respectively. The adjusted R^2 and predicted R^2 show reasonable agreement, with the difference being less than 0.2 (Anderson & Whitcomb, 2017). Furthermore, this can be verified graphically in Fig. 3, where the values predicted vs experimental plot indicate a good correlation between both values. Therefore, it can be concluded that there is good agreement between these values and that the model can be applied successfully, as suggested by (Abdulredha et al., 2020; Nazir et al., 2017).

The best-fit quadratic equation for the response surface optimisation of the ageing of sugar cane spirit obtained from a regression analysis can be described as follows:

$$TPC = 91.7 - 1.8A + 1.6B + 0.002AB + 0.026A^2 - 0.034B^2 \quad (2)$$

This equation, expressed in terms of actual factors, can be used to make predictions about the TPI within the original units and the range of levels selected for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the centre of the design space.

3.4. Optimising the temperature and UEA time of the ageing of sugar cane spirit by CCD

As mentioned above, to optimise the accelerated ageing of sugar cane spirit at a laboratory scale, RSM was employed. RSM is a powerful statistical tool, which allows one to estimate multiple parameters and their interactions using a mathematical model (Kumar et al., 2021). Furthermore, it has the advantage of a decreased number of experimental trials required. CCD is an RSM design commonly applied for the optimisation and evaluation of extraction processes of bioactive compounds, such as in the optimisation of the carotenoid extraction from buriti pulp (*Mauritia flexuosa*) (Silva et al., 2022), total phenolic content of *Opuntia-ficus indica* [L.] Mill. flower (Brahmi et al., 2022), optimisation conditions for the extraction of basil seed mucilage (Nazir et al., 2017) and optimisation of the recovery of phenolic compounds from residual wastewater from table olives (Cazares-Carrión & Abreu-Naranjo, 2021). As seen in Fig. 4, the temperature and UEA-t variables and their relationship with TPI were examined using CCD.

Experimental values for TPI varied from 73.5–86.7 mg GAE_{eq}./L. for the 19 different conditions of ageing using UAE. The response surface plots clearly show that the TPI increased by increasing the temperature and sonicated time for the range of values selected for each factor. A relatively low TPI was obtained at temperatures lower than 45°C and exposure times to ultrasonic waves of below 13 min, as seen in Fig. 4 (A and B). The highest values of TPI (> 84 mg GAE_{eq}./L) resulted from a high temperature (47°C) and UEA time (18 min). However, previous studies have reported a decrease in the extraction yield of phenolic compounds at higher temperatures (Abreu-Naranjo et al., 2018). Another disadvantage that occurs when high temperatures are applied is a reduction in the extraction rate constant, due to the decrease in the intensity of cavitation, associated with a lower surface tension and an increase in the vapour pressure of the cavitation bubbles (Medina-Torres et al., 2017; Tao et al., 2014).

Additionally, numerical optimisation was used to determine the optimal conditions. A value of 85 GAE_{eq}./L, comparable to the Solera Reserve brandy reported by Muñoz et al. (2020), was the objective selected as the optimal condition. Design-Expert predicted the temperature and sonication time parameters of 48.9°C and 18.8 min, respectively, to reach the selected TPI value.

4. Conclusions

The combination of white oak chips and ultrasound energy as an accelerated ageing method for sugar cane spirit had a positive effect, increasing the content of polyphenols. On the other hand, toasting the chips had a negative influence, decreasing the TPI in the aged samples compared to those not subjected to thermic pre-treatment. A direct relationship between intensity of colour (lowest L*) and TPI was observed. Therefore, the ageing treatment applied produces a sugar cane spirit with a more intense colour. The statistical parameters (ANOVA) indicated that the model is adequate to represent the experimental data, with a good agreement between these values and the predicted values for the model. RSM type CCD was successfully employed to optimise the ageing conditions (48.9°C and 18.8 min UAE-t)

to obtain a TPI value of 85 GAE_{eq}./L, comparable to Solera Reserve brandy aged between 12 to 18 months by the traditional method, but in less time. However, additional studies are needed to increase understanding of other effects associated with this ageing method on sugar cane spirit's quality and to delve into other organoleptic properties.

Declarations

Author Contribution Statement

R.A-N: conceptualisation, analysed and interpreted the data, writing - original draft, review & editing, supervision. E.G.Y: performed the experiments, analysed and interpreted the data, review & editing. M.R: performed the experiments, analysed and interpreted the data, review & editing. L.S.: analysed and interpreted the data, review & editing. A.P.M: conceptualisation, methodology, analysed and interpreted the data, review & editing. All authors reviewed and approved the final version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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Figures

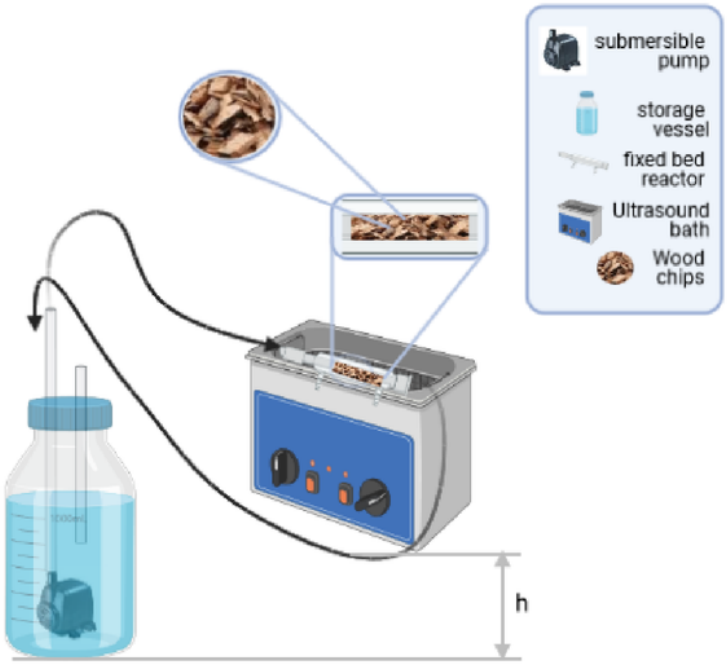


Figure 1

Figure 1

Scheme used for the accelerated ageing of sugarcane spirit using the UAE method

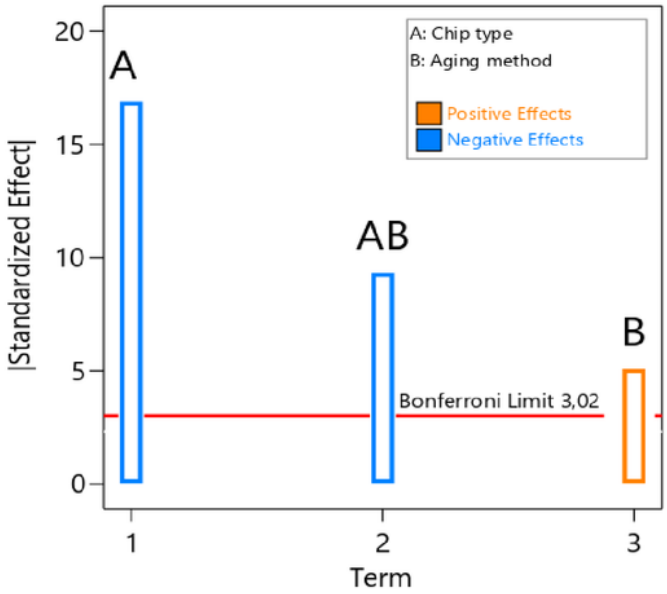


Figure 2

Figure 2

Pareto chart of the effect of each factor and its influence on the TPI

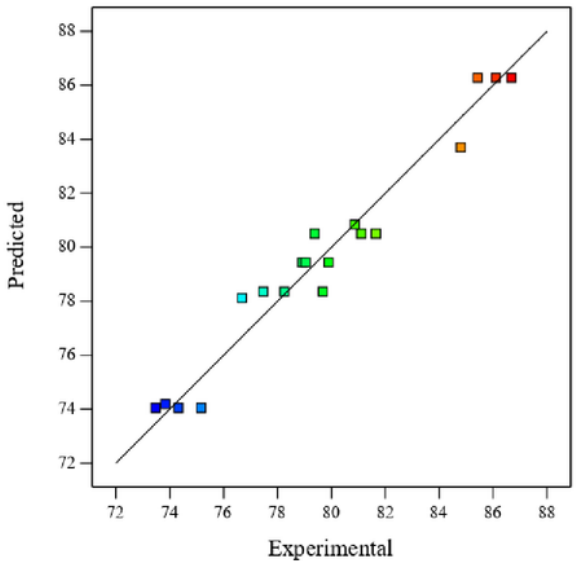


Figure 3

Figure 3

Relationship between predicted and actual values of TPI obtained in the accelerated ageing process of sugar cane spirit using UEA

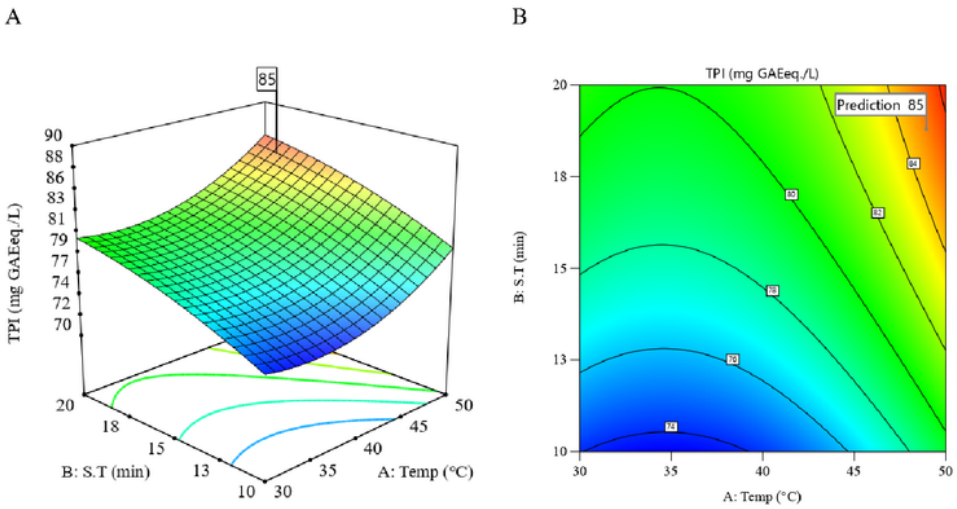


Figure 4

Figure 4

Response surface plot 3D surface (A) and contour (B) of TPI (mg GAEeq./L) of the optimisation of the accelerated ageing of sugar cane spirit