

0.999; $R^2_{Adj Y_3} = 0.987$). The statistical results obtained for the dependent variables (Y_1 – Y_3) suggest that the model is appropriate for the data, as most of the factors and the interactions considered in the experimental design were significant at $p < 0.05$.

3.4. Statistical Analysis of Coefficients

The significance of each coefficient was determined by P-values which were listed in Table 4. The ANOVA analysis of the optimization study indicated that X_1 , X_2 , X_1^2 and X_2^2 were more significant ($P < 0.001$) than the effect of the interaction between the two independent variables (X_{12}). Mixing time and flowers quantity had a positive effect on γ -terpinene, p -cymene and carvacrol retention yields. However, the coefficient β_1 (0.0252, 0.0322 and 0.0025, for Y_1 , Y_2 and Y_3 , respectively) was lower than β_2 (0.0733, 0.1075, 0.00750 for Y_1 , Y_2 and Y_3 , respectively) at the $P < 0.001$ probability level, therefore the factor X_2 (Flowers quantity) was the most important factor affecting γ -terpinene, p -cymene and carvacrol retention by corn oil while the overall effect of X_1 was negligible because of the negative quadratic effect obtained ($\beta_{11} > 0$; $\beta_{11} < 0$). Hypothesis of no global significance factor X_1 , is verified by examining response, that are well spread along the horizontal axis representing X_1 . Negative quadratic effect was also obtained for flowers quantity at the $P < 0.001$ probability level ($\beta_{22} < 0$). This effect gave the appearance of curved curvilinear response surface to have optimal conditions for each response. The coefficient β_{12} of X_1X_2 interaction was found to be not significant ($P > 0.05$) for Y_1 and Y_3 . Conversely, positive quadratic effects were obtained for mixing time and flowers quantity at the $P < 0.001$ probability level for Y_3 ($\beta_{12} = 0.033$, at $P < 0.001$)

The final equations for optimization of corn oil aromatization parameters derived from the application of the method (after eliminating non-significant terms) are given below:

$$Y_1 = 0.1691 + 0.0252 X_1 + 0.0733 X_2 - 0.0202 X_1^2 - 0.0477 X_2^2 + 0.0042 X_1X_2 \quad (2)$$

$$Y_2 = 0.2392 + 0.0322 X_1 + 0.1075 X_2 - 0.0404 X_1^2 - 0.0304 X_2^2 + 0.0330 X_1X_2 \quad (3)$$

$$Y_3 = 0.0169 + 0.0025 X_1 + 0.0075 X_2 - 0.0018 X_1^2 - 0.00487 X_2^2 + 0.0002 X_1X_2 \quad (4)$$

3.5. Interpretation of the Response Surface Model

The relationship between the responses and the experimental variables can be illustrated graphically by plotting three-dimensional response surface plots (Fig. 2). The vertical axes show γ -terpinene, p -cymene and carvacrol retention yield (Y_1 , Y_2 and Y_3 , respectively), and each of the two horizontal axes represents the two independent variables (mixing time (X_1) and flowers quantity (X_2)). The topography of these response surfaces are also illustrated by isoresponse contours representing lines of constant response in a two variable plane. Such plots are helpful in studying the effects of the variation of the factors in the domain studied and consequently, in determining the optimal experimental conditions [35]. In Fig. 2, the examination of the isoresponse contours and three-dimensional plots showed that γ -terpinene, p -cymene and carvacrol retention by corn oil

increased when increasing flowers quantity and mixing time. Sample presenting the most significant γ -terpinene, p -cymene and carvacrol contents (0.202 mg/ml, 0.341 mg/ml and 0.020 mg/ml, respectively) is that containing the highest flowers amount (5 g) and flavouring for the longer period of mixing time (25 min).

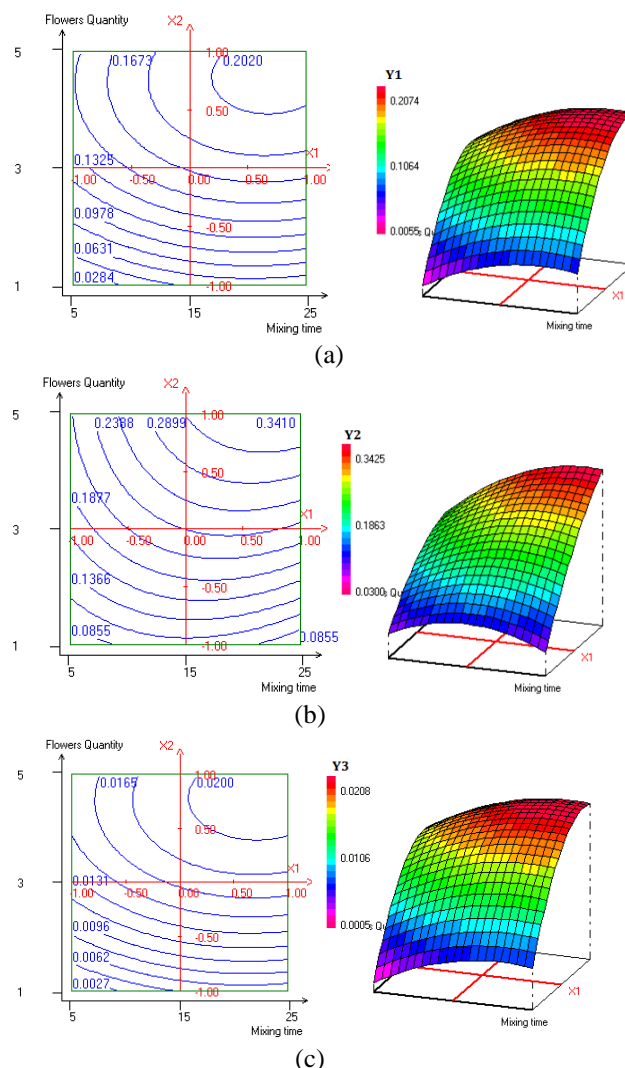


Figure 2: Three-dimensional response surface and contour plots for the effect of incubation time and peel quantity on γ -terpinene (a), p -cymene (b) and carvacrol (c) retention in refined corn oil.

Optimal conditions selected by the software NEMRODW were: Mixing time: 25 min and flowers quantity: 5 g. Under these conditions, the expected values of the γ -terpinene, p -cymene and carvacrol yields were $Y_{1op} = 0.20403$ mg/ml, $Y_{2op} = 0.34102$ mg/ml, and $Y_{3op} = 0.02057$ mg/ml, respectively.

4. Conclusion

This work has revealed that monoterpene hydrocarbons were abundant in the flavoured oil forming 92.59 % of the total aroma mainly represented by p -cymene, α -thujene and γ -terpinene. Thyme flowers major volatiles were carvacrol, γ -

terpinene and *p*-cymene. Due to the several biological activities of these volatile compounds, the response surface methodology was a useful tool to determine the optimal experimental conditions of their retention by refined corn oil. Aroma retention by refined corn oil increased significantly with increasing peel quantity and mixing time. The selected optimal conditions (Mixing time: 25 min and flowers quantity: 5 g) have been checked and confirmed. This enriched oil may have important biological activity. In fact, thyme EO and its volatile compounds exhibit a range of biological activities. A future study will focus on the influence of the γ -terpinene, *p*-cymene and carvacrol retention on phenolic composition of the flavoured corn oil and its antioxidant activities.

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