Optimisation of Microwave-Assisted Extraction of Date Seed Oil

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Abstract

Background: Date seed waste (DSW), a byproduct of the date industry, can extract high-value oils. Using this biomass improves food industry sustainability. **Aim:** This study aimed to optimise microwave-assisted extraction (MAE) parameters to maximise the oil yield from DSW. **Method:** An optimisation approach was used for one-factor-at-a time (OFAT). The study examined the impact of microwave power (400–800 W), extraction time (10–20 min), and solvent volume (80–120mL) on oil yield. A second-order polynomial regression model predicted the extraction outcomes to ensure statistical reliability, and they performed each experiment 3 times. **Results:** DoE analysis identified extraction time ($F=20.96$, $p \leq 0.0001$) and microwave power ($F=13.56$, $p \leq 0.0001$) as the most significant factors, whereas solvent volume had a lesser effect ($p \geq 0.05$). The model demonstrated a high predictive accuracy ($R^2=0.90$). Optimal extraction conditions, which used 700 W microwave power, 15 min extraction time, and 80 mL solvent volume as H_2O, achieved a validated maximum oil yield of 9.14 ± 0.124%. The prediction error was

4.6%, and validation showed no difference in yield ($p \geq 0.05$). **Conclusions:** The MAE protocol effectively extracted oil from DSW and exhibited superior predictive capability compared to OFAT. **Recommendation:** The DoE-optimised MAE protocol enables sustainable oil recovery from DSW, supporting valorisation and efficiency.

Keywords: Seed oil Yield %, Extraction time (T), Microwave power (W), Solvent Type (mL), Multilevel Categoric, and One-Factor-at-a-Time Approach

1. Introduction

The worldwide necessity to transition from a linear "take-make-dispose" economic model to a circular economy has prompted a fundamental change in how industries view waste (Oladzad *et al.*, 2021). Agricultural by-products are now recognised as abundant and renewable resources for producing high-value biochemicals, biofuels, and functional materials, rather than environmental and economic burdens (Hagman and Feiz, 2021). Valorisation is the primary method for achieving waste minimisation, reducing fossil fuel reliance, and generating bio-economy revenue (Rana *et al.*, 2020).

Date palms (*Phoenix dactylifera L.*) offer a substantial opportunity (Shukla *et al.*, 2020). Each year, the global fruit production industry produces millions of tonnes of fruit and generates billions of dollars in revenue (Hammami *et al.*, 2024). As a result, many by-products are produced, and date seeds make up roughly 10-15% of the fruit's weight (Tlais *et al.*, 2020). Traditionally, these date seeds have mostly been wasted or given to animals as poor-quality feed, which has created environmental problems due to their disposal (Muñoz-Tébar *et al.* 2024). However, date seeds contain valuable biochemicals, notably lipids, carbohydrates, proteins, dietary fibres, and significant antioxidant phenolic compounds (Mrabet *et al.*, 2020, Najjar *et al.*, 2020). Hence, strategically converting this underused biomass into valuable goods is vital for making the date sector more sustainable and economically viable (Oladzad *et al.* 2021).

Date seed oil, a lipid component of date seeds, has gained considerable scientific and commercial attention (Abdalla *et al.*, 2012). It contains considerable amounts of unsaturated fatty acids, namely oleic acid, and unique bioactive compounds, making it suitable for functional food ingredients, cosmetics, nutraceuticals, and biofuel production (Mrabet *et al.*, 2020). However, the main problem is creating an extraction method that is yield-efficient and green, reducing energy use, limiting dangerous solvents, and speeding up time (Pappas *et al.*, 2021).

Traditional techniques, such as Soxhlet extraction, despite their effectiveness, are often associated with extended extraction periods, high solvent use, and potential heat-induced breakdown of sensitive components (Gori *et al.* 2020). To address these constraints, advanced and unconventional extraction techniques have been developed (Chaves *et al.*, 2020). MAE is a promising green alternative (Cai *et al.*, 2013; López-Salazar *et al.*, 2023; Nour *et al.*, 2021; Ondruschka and Asghari, 2006; Petrotos *et al.*, 2021). MAE involves direct dielectric heating, wherein the polar molecules in the plant absorb energy (Zhang *et al.*, 2011). Quick, focused heating causes pressure changes that break down cell walls, enabling the rapid movement of cell contents into the solvent (Ondruschka and Asghari, 2006). MAE surpasses conventional approaches because it provides shorter extraction times (minutes rather than hours), decreased solvent usage, increased extraction outputs, enhanced product purity, and improved energy conservation, rendering it an excellent choice for the eco-friendly processing of biomass, such as date seeds (Ondruschka and Asghari, 2006).

For any MAE process to be effective, tight control and optimisation of key operating parameters are necessary (Sui, 2025), including microwave power, extraction time, solvent type, and solvent-to-solid ratio (López-Salazar *et al.*, 2023). MAE has various advantages over traditional extraction methods, especially Soxhlet extraction, including reduced extraction times and solvent consumption and increased extract yields. These advantages help reduce operational costs (Danlami *et al.*, 2014). The industrial scaling of MAE presents several challenges because the outstanding results achieved on the laboratory scale regarding energy and cost reductions are not always true at larger scales. Such discrepancies are usually justified by the complexity of the equipment design and integrity issues of sensitive reactive intermediates upon longer microwave radiation (Rosa *et al.*, 2018, Voss *et al.*, 2023). MAE can be competitive in value addition to agricultural by-products, especially at larger plant scales and with low-cost feedstocks; however, the capital investment for industrial-scale MAE is significant (Herrero and Ibáñez, 2017). Hence, despite the expected potential of MAE to be more efficient and eco-friendly (i.e. low energy consumption and waste generation), its usefulness in large-scale oil recovery requires careful investigation of its scalability and economic aspects to guarantee its viability (Aquino *et al.*, 2023).

Owing to the complex and often nonlinear interactions between these factors, optimising the process for maximum yield requires a statistically robust and systematic strategy (Bhan *et al.*, 2016). The OFAT approach is a common and traditional method for chemical engineering process optimisation (Mannino 2015). A single factor was changed to assess its influence on the results, whereas the others remained constant (Singh *et al.*, 2011). Despite its simplicity and intuitiveness, the OFAT method is fundamentally flawed because it does not consider the interactions between variables (Shana *et al.*, 2023, Singh *et al.*, 2023). For example, the optimal extraction time might vary depending on the microwave power. OFAT is inefficient because it ignores synergistic or antagonistic effects, requires many experimental runs, and does not determine the real global optimum of the process (Petrotos *et al.*, 2021).

Statistical DoE methods, including Response Surface Methodology (RSM) and Multilevel Categoric designs, have become the go-to methods for process optimisation because they can surmount these limitations (G and Prabaharan, 2020, Elnour *et al.*, 2018). By allowing the structured, simultaneous change of multiple factors, the DoE allows one to quantify both the main effects and their complex interactions (Carbone *et al.*, 2023). With this approach, the process is fully understood, fewer experiments are necessary, and a predictive mathematical model can be produced to link the factors and responses of the process (St-Pierre and Weiss, 2009). This model facilitates space exploration and pinpoints the ideal operating conditions with a strong statistical assurance (Sahu *et al.*, 2017).

MAE's use of MAE for extracting bioactive compounds from plants is well documented, and the theoretical advantages of DoE over OFAT are widely accepted; however, a direct comparison of these methods for optimising oil extraction from DSW is lacking (Haffizi *et al.*, 2020). Current research frequently applies a single method or focuses on various biomass sources (Juhaimi *et al.*, 2019). A comprehensive comparative analysis is required to empirically highlight the accuracy and outcome variances when the OFAT and a complex multilevel categorical DoE are used on the same system (Mishra *et al.*, 2022).

This study is based on a two-fold rationale. First, it seeks to methodically improve the MAE process to increase the output of beneficial oil from discarded date seeds, thus offering an applicable solution for industrial waste reuse (Jibril *et al.*, 2020). Second, it aims to provide an evidence-based comparison of OFAT and DoE optimisation, showing the limits of OFAT and the predictive ability of the latter (Antony *et al.*, 2003). Researchers and engineers can use this comparative analysis as a case study to optimise green extraction from different biomass sources (Tobin *et al.*, 2020).

This study investigated the efficient extraction of oil from date seeds, focusing on 3 key areas. First, it rigorously explores how microwave power, solvent volume, and extraction time interactively and independently influence oil yield. Second, it methodically identifies the optimal combination of these parameters to maximise the oil yield. This study thoroughly compared the optimised approach with the traditional OFAT method, evaluating both the outcomes and efficiency. The core aim of this study was to establish and refine an MAE process to maximise oil recovery from date seeds. Initially, the OFAT method was employed to effectively screen the MAE parameters and define the crucial operating range (López-Salazar *et al.*, 2023). Subsequently, a multilevel categorical DoE was used to assess the significant effects and interactions of microwave power, solvent volume, and extraction duration on oil yield. The DoE data provided the foundation for developing a precise mathematical model to predict oil yield based on the MAE parameters (Nour *et al.*, 2021). This model helped determine and validate the MAE conditions that yielded the highest oil production. The final stage involved a comprehensive comparison of the OFAT and DoE methodologies in terms of their results, efficiencies, and predictive capabilities.

This study is based on several critical hypotheses grounded in fundamental chemical engineering principles and statistical theories(Gilman *et al.*, 2021). We predicted that a DoE statistical model would forecast date seed oil yield and that the identified optimal conditions would be validated through experiments. Furthermore, we anticipated that microwave power and extraction time would significantly influence oil yield, as suggested by the initial ANOVA results, which showed p-values below 0.05 for both factors (Nyam & Lau, 2015). Ultimately, we contend that the DoE is a superior optimisation tool that focuses on the OFAT approach (Petrotos *et al.* 2021). This superiority stems from the DoE's capability to capture intricate variable interactions, leading to the identification of true optimal conditions, which is a significant limitation of the OFAT method that hinders its ability to achieve genuine optimisation (Czitrom, 1999).

2. MATERIALS AND METHODS

2.1 Materials

The extraction process used several key materials and pieces of equipment. Crushed date seed powder (10 g per extraction) was used as the raw material, and distilled water was used as the extraction solvent. Primary extraction was performed using an Ethos reflux microwave extractor (Milestone, Italy) controlled by easy-control software for 3-level parameter management. Post-extraction processing involved a vacuum pump for filtration and a rotary evaporator (Buchi Rotavapor R-200 coupled to Buchi Vac V500 pump, Switzerland) for determining the sample concentration. The experimental design and parameter optimisation were conducted using Design Expert 13 software (version 13; Stat-Ease Inc., Minneapolis, USA).

2.2 Sample collection and preparation

Samples of Hilwa Al-Jouf, Ajwa Al-Madinah, Sukari Al-Qassim, and Khalas Al-Ahsa dates (2025 season) were crushed into a powder, as shown in **Fig 1.** For the OFAT experiments, a precisely weighed 10 g sample of the powder was mixed with distilled water in quantities specified by the experimental design. According to Alara *et al.* (2018), the mixture was loaded into a microwave extractor (Ethos reflux microwave extractor, Milestone, Italy) for the subsequent extraction procedures. The extraction parameter conditions were set according to the experimental values generated by the Multilevel Categoric Design-of-Experiment; One and OFAT used

Design Expert 13 software (version 13, Stat-Ease Inc., Minneapolis, USA), as listed in **Table 1.** The microwave extraction process used a three-level heating protocol designed to achieve a noise reduction and temperature control. The heating sequence consisted of: (1) pre-heating for 2 min at the extraction temperature, (2) irradiation at the extraction temperature according to the experimental design, and (3) cooling to 30°C for 2 min. A pulsed heating approach (preheating-irradiation-cooling modes) was implemented to reduce the noise factor imposed by temperature fluctuations on the microwave-assisted extractors. The microwave oven was operated stepwise using the 'easy-control' software programmed for 3-level control of the irradiation time, microwave power, and temperature parameters. Following microwave irradiation, the sample was filtered using a vacuum pump to separate the liquid extract from the solid sample residue. The filtered extract was subsequently concentrated using a rotary evaporator (Buchi Rotavapor R-200 coupled to Buchi Vac V500 pump, Switzerland) to remove excess solvent. The resulting crude extract was stored under refrigeration at 4°C until further analysis. The entire extraction process was performed in triplicates to ensure reproducibility and statistical validity of the results.

2.4 Experimental design

2.4.1 Experimental design of OFAT

Using the traditional "OFAT" method for optimisation, researchers investigated the influence of date seed oil yield%, extraction time (T), microwave power (W), and solvent volume (mL). The extraction methodology employed 3 critical process variables that significantly influenced the extraction yield and efficiency. The microwave power served as the primary energy input parameter, with values ranging from 400 W to 800 W to provide adequate heating while preventing thermal degradation of the target compounds. The volume of the solvent (distilled water) was varied between 79.9 mL and 119.9 mL to optimise the solid-to-liquid ratio and ensure complete sample immersion while maintaining cost-effectiveness. Researchers controlled the extraction time between 9.9 and 20 min to balance extraction completeness with processing efficiency and prevent overheating of thermolabile compounds.

2.4.2 Multilevel categoric design (MCD)

The MCD concept was employed to illustrate the nature of the response surface in the experimental region and elucidate the optimal concentrations of the most significant independent variables. This model included 3 variables: extraction time (T), microwave power (W), and solvent volume (mL). Based on the "OFAT" method results, they fixed the other parameter, that is, yield %. Table 1 shows how the factors were examined at 5 different levels (relatively low, low, basal, high, and relatively high) coded (-2, -1, 0, +1, + 2). Based on the MCD for the 3 variables, 125 experimental runs were executed, and their observations were fitted to second-order polynomial model. where yield is the dependent variable (date seed oil productivity), and A, B, and C are the independent variables (extraction time (T), microwave power (W), and solvent volume (mL)). The regression coefficient at the centre point; +0.4218 and 0.0132 are the linear coefficients. Table 1 presents the percentage yield of seed oil from date waste extracted using MAE under varying conditions, based on the MCD experiment for response on-factor- at-time analysis.

3. Results and Discussion

3.1 Optimum levels of parameters determined by OFAT method

Fig. 1 presents the influence of extraction time on date seed oil yield, showing an optimal duration of 15 min to maximise oil production. A significant reduction in yield was observed when the extraction time was less than 9 min or more than 20 min. The duration of oil extraction significantly impacts the amount of oil extracted from the seeds. Limited extraction duration hinders effective solvent permeation into the dense seed matrix, impeding the solubilisation of oil compounds and leading to incomplete oil extraction. These findings align with those of Gul *et al.*'s study on slow diffusion in dense matrices (2022).

Conversely, extending the extraction time to 15 min diminished the oil yield. While longer durations typically enhance solute transfer, prolonged microwave exposure likely degrades thermolabile oil components (Andriana and Broto, 2023). Furthermore, elevated temperatures from extended irradiation can adversely affect solvent properties and compromise solute stability, which explains the observed reduction (Dimić *et al.*, 2021). Although previous studies have broadly examined extraction parameters, determining the optimal duration for date seed oil extraction is crucial (Pao-la-or *et al.*, 2023). The optimal extraction duration was determined to be 15 min, which maximised the oil yield and prevented degradation due to extended processing. An 80 mL solvent volume and a 15-minute extraction time yielded the highest date seed oil yield. The amount of distilled water was a crucial factor, with yields dropping significantly when outside the 79-100 mL range. This decrease at lower volumes is likely due to an inadequate solvent-to-seed ratio, which hinders complete oil compound dissolution and seed matrix penetration (Dimić *et al.* 2021; Gul *et al.* 2022). Increasing the solvent volume beyond 100 mL notably reduced the extraction yield. This is primarily due to mass transfer limitations, as the increased dilution of the extract phase diminishes the concentration gradient between the seed matrix and solvent, thereby weakening the thermodynamic driving force for oil diffusion (Dimić *et al.*, 2021, Pao-la-or *et al.*, 2023).

The optimal extraction time was determined to be 15 min. Shorter durations may not allow sufficient solvent penetration or extraction of the target compounds. Extending the extraction time beyond 20 min did not yield a statistically significant increase in recovery and could potentially lead to the degradation of sensitive compounds or loss of volatile oil components due to prolonged exposure to the extraction conditions (Gul *et al.*, 2022, Ramkumar *et al.*, 2014). The most effective parameters for extracting oil from date seeds were identified as using 80 mL of solvent for a 15-minute extraction period. This configuration ensures an optimal solvent-to-seed ratio, which enhances both mass transfer and solubility while preventing potential degradation of product quality due to over-dilution or extended processing times. Additionally, the study identified 700W as the ideal microwave power setting for maximising the yield of date seed oil. Deviations from this optimal power, particularly reductions below 600 W or increases above 800 W, resulted in a significant decline in oil yield, highlighting the crucial role of microwave power in governing extraction efficiency and maintaining the quality of the extracted oil. Insufficient microwave power, below 600 W, was inadequate for efficient oil extraction.

Suboptimal oil extraction from date seed waste (DSW) is largely due to insufficient heating, which cannot adequately overcome the natural resistance of the seed matrix, thereby preventing the release of oil compounds (Gul *et al.*, 2022; Dimić *et al.*, 2021). Furthermore, an inadequate energy input at reduced power levels diminishes the solvent's ability to infiltrate the seeds and dissolve the oil effectively. This compromises the essential interactions between the solvent and seeds, as well as the structural changes within the seed matrix, which are necessary for efficient oil recovery (Hosokawa *et al.*, 2004; Ramkumar *et al.*, 2014).

Conversely, microwave power exceeding 800 W is empirically linked to a notable decline in oil yield. This reduction is primarily attributed to the elevated susceptibility to thermal degradation of the matrix. Such intense thermal stress can precipitate detrimental processes, including undesirable chemical alterations, accelerated oxidative reactions, and premature volatilisation of valuable lipophilic components (Pao-la-or *et al.*, 2023, Andriana and Broto, 2023). Moreover, excessively high power levels present a significant risk of disrupting the critical solvent-seed interactions essential for effective extraction. Such conditions can instigate adverse structural modifications within the complex date seed matrix, thereby hindering the optimal release and subsequent recovery of oil (Shimojo *et al.*, 2020; Lawton and Budil, 2009). These combined factors underscore the importance of optimising microwave power to prevent detrimental effects on the extraction efficiency and product quality. The reduction in yield observed at both the lower and upper limits of the utilised power spectrum was predominantly due to the characteristic selective heating mechanism of microwave irradiation. This phenomenon can generate localised thermal gradients and a non-uniform temperature distribution throughout the sample, potentially compromising the overall extraction efficiency (Valenta *et al.*, 1997; Young *et al.*, 2002). Such temperature variations present a considerable obstacle, potentially resulting in less-than-ideal extraction performance in cooler zones or, conversely, exacerbating thermal degradation in hotter regions.

These adverse conditions collectively contribute to a reduction in the ultimate yield of extraction. Consequently, the identification of the optimal microwave power (700 W) is paramount. This power level establishes a precise equilibrium, facilitating efficient extraction while simultaneously mitigating the risks of thermal degradation and preserving the structural and interactive integrity of the date seed matrix. **Fig 1** shows the impact of microwave power (W) on the extraction yield (%). **Fig 2** shows the **intricate interactions** among extraction time (min), solvent volume (mL), and microwave power (W), as determined by the one-factor-at-a-time method. **(Figure 1 and Figure 2 locations).**

3.2 Optimisation of process parameters using multilevel categoric DoE

The process conditions, including extraction time (min), microwave power (W), and solvent volume (mL), were systematically optimised as independent variables to achieve the maximum oil yield percentage from the DSW. This optimisation was conducted using a multilevel categorical design-of-experiment approach to evaluate the individual and interactive effects of each parameter on the extraction efficiency. The regression model for extracting date seed oil was statistically significant, according to the ANOVA results in **Table 2**. The model was statistically significant at the 99% confidence level, as indicated by its high significance ($F = 4.66$, $p < 0.0001$; $p < 0.01$). The date seed oil yield appears to be meaningfully impacted by these independent variables as a group.

The analysis showed that the individual parameters had noticeably different importance patterns. Microwave power (A) showed high statistical significance ($F = 13.56$, $p < 0.0001$, $p < 0.01$), demonstrating its critical importance in extraction efficiency. The F-value of 20.96 and p-value < 0.0001 ($p < 0.01$) for extraction time (C) demonstrated its highest significance, suggesting that it was the most impactful parameter among the 3 factors investigated.

In contrast, the F-value of 2.17 and p-value of 0.0823 ($p > 0.05$) suggest that solvent volume (B) is non-significant, demonstrating that oil yield is not significantly affected by solvent volume variations in the tested range. However, this does not explain the model's performance, as the overall model shows an excellent fit. With an F-value of 1.73, the lack-of-fit test p-value > 0.05 was non-significant, indicating that the model adequately described the data without requiring more complex models or additional factors. With an R-squared value of 0.90, approximately 90% of the total variation in the date seed oil yield was accounted for by the model, with only 10% being unaccounted for. The non-significant contribution of the solvent volume did not substantially affect the overall model performance owing to the strong effects of the other parameters and their interactions. An adjusted R-squared of 0.706 suggests that the regression model is robust for optimising date seed oil extraction, where microwave power and extraction time are key variables, along with their significant interactions to maximise yield. With 188 degrees of freedom for the corrected total, the total sum of squares (11.544) represented the baseline variation, and the unexplained variation was represented by the residual sum of squares (1.154).

The comprehensive analysis revealed significant interactions between the process parameters. The AB interaction (microwave power × solvent volume) was statistically significant ($F = 3.24$, $p < 0.0001$), indicating that the effect of microwave power on the oil yield depended on the solvent volume. Similarly, the AC interaction (microwave power × extraction time) was highly significant ($F = 4.75$, $p < 0.0001$), suggesting synergistic effects between these 2 critical parameters. The BC interaction (solvent volume × extraction time) was also significant ($F = 3.31$, $p < 0.0001$), despite the solvent volume being non-significant as the main effect. Notably, the three-way interaction ABC ($F = 3.90$, $p < 0.0001$) was highly significant, indicating complex interdependencies among all 3 factors that must be considered for optimal extraction conditions.

Interestingly, the comprehensive design matrix, encompassing both coded and actual values of the variables, is presented in **Table 1**, along with the corresponding experimental and predicted results for date seed oil production. The predicted values were calculated using equation **2** to provide a framework for the model validation. All extraction experiments were performed in triplicate to ensure statistical reliability and reproducibility of the results. To mathematically predict the optimal extraction conditions within the established experimental constraints, a multilevel categoric model was fitted to the experimental data of date seed oil production. Mathematical modelling was accomplished using Design Expert software, which facilitated the development of a comprehensive factorial design for process optimisation and enabled the identification of optimal operating conditions for maximising the oil yield.

Yield % = $\beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 A^2 + \beta_5 B^2 + \beta_6 C^2 + \beta_7 AB + \beta_8 AC + \beta_9 BC + \varepsilon$ = [Equation 2]

Where:

The dependent variable, *"**Yield %**"* represents the date seed oil yield percentage, which is the primary response targeted for prediction or optimisation. The model intercept, denoted **as** $\beta_0 = 1.75$, establishes the baseline logarithmic yield when all the coded factors are set at their respective reference levels. The direct influence of individual factors on the yield was quantified using the linear main effect coefficients, specifically $\beta_1 A$ through $\beta_4 A$ for factor A, $\beta_1 B$ through $\beta_4 B$ for factor B, and $\beta_1 C$ through $\beta_4 C$ for factor C. Furthermore, the model incorporates various two-way interaction terms to capture the synergistic or antagonistic effects of the factors. For instance, $\beta_7 AC$ signifies a linear-by-linear interaction between factors **A** and **C**, whereas $\beta_8 A^2 C$ and $\beta_9 AC^2$ represent quadratic-by-linear and linear-by-quadratic interactions, respectively. These terms are crucial for elucidating how the impact of one factor is modulated by the level of another.

Beyond two-way interactions, the model also accounts for three-way interaction effects, such as $\beta_{ijk} \cdot A_i B_j C_k$. The higher-order terms capture the combined influence of all 3 factors, demonstrating how the interaction between any 2 factors is modified by the third. A random error term (ε) was included to account for all the unexplained variability within the model. A comprehensive model architecture was meticulously engineered to capture the complex nonlinear relationships between process variables and the observed yield, addressing potential measurement inaccuracies, the influence of unmeasured or uncontrolled factors, and the inherent variability of natural processes. This approach is advantageous for optimisation studies in which intricate factor interactions are recognised as affecting the response variable.

A thorough regression analysis was performed to optimise the parameters for date seed oil production, revealing a complex correlation between the process variables and the resulting oil yield. Specifically, the oil yield percentage was established as a multifaceted function influenced by microwave power (A), solvent volume (B), extraction duration(C), and the intricate interactions among these 3 factors. The coefficient of determination (R^2) of 0.90 for the developed date seed oil production model highlighted a robust and significant relationship between the experimentally measured oil content and the model's predicted values, indicating a key indicator of the model's predictive capability. The coefficient suggested that the model accounted for 90% of the total variability in the date seed oil yield. Therefore, only 10% of the overall variation in oil production was unexplained by the current model parameters, indicating a high level of predictive accuracy and explanatory power for the regression Equation within the experimental range.

The high $\mathbf{R^2}$ value indicates that the developed model accurately represents the correlation between the independent variables and date seed oil yield, encompassing intricate parameter interactions. This strong model fit results from a thorough factorial design that accounts for both primary and interactive effects during the extraction process. **Table 2** provides additional statistical information, including the model significance and contributions of individual parameters within the ANOVA results. The regression model exhibited substantial statistical significance, as confirmed by the ANOVA.

Fisher's F-test yielded a probability value (p-model > F) of less than 0.0001. The model's F-value of 4.66 shows that the observed significance is unlikely to be due to random noise, with less than a 0.01% chance of such an occurrence. The complete ANOVA results are presented in Table 2. The individual parameter analysis revealed that the linear terms for microwave power (A) and extraction time (C) were the most significant among all major effects, both showing p-values of < 0.0001. P-values less than 0.0500 indicated that the model terms were significant at a 95% confidence interval. Therefore, microwave power (A) and extraction time (C) were identified as the most significant main effect terms affecting date seed oil yield, whereas solvent volume (B) showed less pronounced individual effects on the extraction process.

The regression model terms for solvent volume (B) were insignificant (p = 0.0823 > 0.05) as a major effect at the 95% confidence level, but this parameter showed significant interactions with other factors. The linear major effects of the significant parameters (microwave power and extraction time) were significant, suggesting that these factors were critical process variables. Variations in their magnitude could revolutionise the yield of date seed oil production. However, the significant interaction effects (AB, AC, BC, and ABC) demonstrate that interactive effects are crucial for achieving true optimisation, rather than relying solely on individual parameter optimisation. This finding emphasises the importance of considering parameter interactions when optimising extraction processes.

In addition to confirming the significance of microwave power (A) and extraction time (C) as the main effect terms, the lack-of-fit test was desirable for establishing the adequacy of the model ($F = 1.73$, $p > 0.05$). The lack-of-fit test serves as a diagnostic tool to determine whether the selected model describes the observed data or whether a more complex model is required to do so. Because the p-value for the lack-of-fit in the ANOVA analysis was greater than 0.05, the model appears to be adequate for representing the observed data at a 95% confidence level. This statistical validation confirmed that the current model structure is appropriate for describing the relationship between the process variables and date seed oil yield. The coefficient of variation (CV = 6.7%) indicated good precision and reliability of the experimental data, whereas the PRESS value of 10.03 suggested reasonable predictive capability of the model for future observations. Finally, the response on-factor at-time ANOVA for the MCD experiment (CDE) is presented in **Table 2.** (Table 2 location)

3.3 Parameter response surface analysis

The results of the yield % response surface analysis of microwave power, solvent volume, and extraction time for the maximum production of date seed oil are presented in **Table 2**. The model term value (p-value <0.0001) indicated a good relationship between the yield % of date seed oil production and the extraction parameters (microwave power and extraction time). In this study, the final equation in terms of the actual factors is presented in Eq. (3). Therefore, the fitting major effect polynomial equation of the data with only significant terms is given by equation (3).

Yield % = $\beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 A^2 + \beta_5 B^2 + \beta_6 C^2 + \beta_7 AB + \beta_8 AC + \beta_9 BC + \varepsilon$ = [Equation 3]

where the variables are as defined in Table (3)

Fig 3 shows the relationship between date seed oil production and extraction parameters. The effects of varying microwave power, solvent volume, and their mutual interactions on the yield % are shown in **Fig 2A.** With increasing microwave power, the yield % increased, but with low or fluctuating solvent velocities. However, the highest yield% was observed at a microwave power of 700 W (level 3) and solvent volume of 80 mL. The effects of microwave power, solvent volume, and their interaction on the yield % of date seed oil are shown in Fig 3B.

Fig 3B shows the effect of varying the microwave power and extraction time on the yield of date seed oil. Extraction times of less than 16 min, increasing the extraction microwave power to 700 W resulted in a high yield percentage of date seed oil. At an overheated microwave power of (800 W) (greater than 700 W), the reverse trend is noticeable. Herch *et al.* (2014) and Jadhav *et al.* (2016) showed that microwave power strongly influences date seed oil yield at extraction times shorter than 16 min. The yield improved with increasing power, peaking at approximately 700 W because of the increased extraction efficiency. However, the yield decreased when the power exceeded 700W, possibly owing to thermal oil decay. Therefore, the best extraction occurred at approximately 700 W and short durations. The effects of microwave power, extraction time, and their interaction on the yield % of date seed oil are shown in **Fig 3C**. **(Figure 3 location)**

3.3 Optimisation and validation of date seed oil extraction conditions

To validate the mathematical model, an experiment was conducted in triplicate using the predicted optimal conditions: microwave power of 700 W, extraction time of 15 min, and solvent volume of 80 mL. The average experimental yield showed a strong correlation with the predicted yield, with no significant difference between the 2 ($p \geq 0.05$) and a prediction error of only 4.6% (**Table 3**). This result confirms that the model parameters are optimal for maximising date seed oil recovery. The final validated conditions are listed in **Table 3**. **(Table 3 location).**

4. Conclusion

This study established the optimal conditions for MAE of oil from DSW. This advancement provides a robust strategy for the valorisation of agricultural by-products, contributing to a circular economy. This study examined the influence of microwave power, extraction time, and solvent volume on oil yield. A hybrid methodological framework was utilised, integrating the established OFAT strategy with a comprehensive multilevel categorical DoE. The study identified the optimal conditions for oil recovery as a microwave power of 700 W, extraction duration of 15 min, and solvent volume of 80 mL. Statistical validation through DoE analysis underscored the significant impact of extraction time ($F=20.96$, $p<0.0001$) and microwave power ($F=13.56$, $p<0.0001$) on the process, with solvent volume having a comparatively minor influence ($p>0.05$). A significant finding highlighted the complex interaction effects among all 3 parameters, which is a critical insight beyond the scope of the OFAT method. This demonstrates the superiority of the DoE approach for comprehensive process optimisation. A highly accurate and statistically significant second-order polynomial model ($R^2=0.90$, $p<0.0001$) was developed for reliable oil yield prediction. Experimental validation confirmed the model's robustness, showing no significant deviation between the predicted and actual yields under the optimised conditions ($p\geq0.05$). This study presents a validated, efficient, and sustainable protocol for extracting valuable biochemicals from DSW, highlighting the enhanced predictive capability and comprehensive insights offered by the statistical DoE over traditional optimisation techniques.

Author contributions (with CRediT details)

AAM, AHN, RHM, and AAME designed the study, performed the literature search, collected and interpreted the data, and drafted the manuscript. MGH participated in the manuscript preparation and revision.

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Conflicts of interest

All authors declared that there is no conflict of interest

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Data availability

All supplementary data have been uploaded with manuscript in the submission system

Abbreviations

DSW	Date seed waste
DoE	Design of Expert
MAE	Microwave-assisted extraction
MCD	Multilevel categoric design
OFAT	One-factor-at-a time
RSM	Response Surface Methodology

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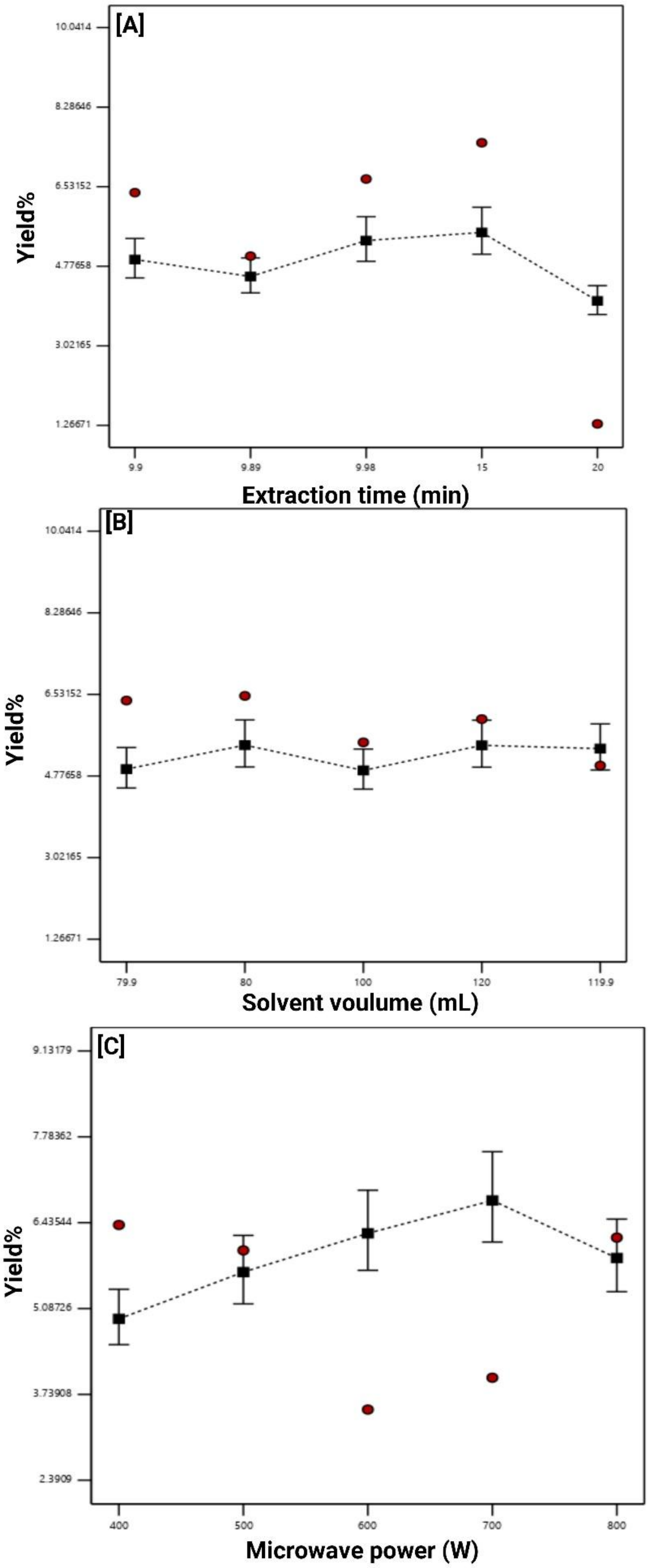

[A]
Yield%
10.0414
8.28646
6.53152
4.77658
3.02165
1.26671
9.9
9.89
9.98
15
20
Extraction time (min)
[B]
Yield%
10.0414
8.28646
6.53152
4.77658
3.02165
1.26671
79.9
80
100
120
119.9
Solvent voulume (mL)
[C]
Yield%
9.13179
7.78362
6.43544
5.08726
3.73908
2.3909
400
500
600
700
800
Microwave power (W)

Fig 1. Impact of yield% on date seed oil output [A] extraction time (min), [B] solvent volume (mL), and [C] microwave power (W) using the One-Factor-At-Time method.

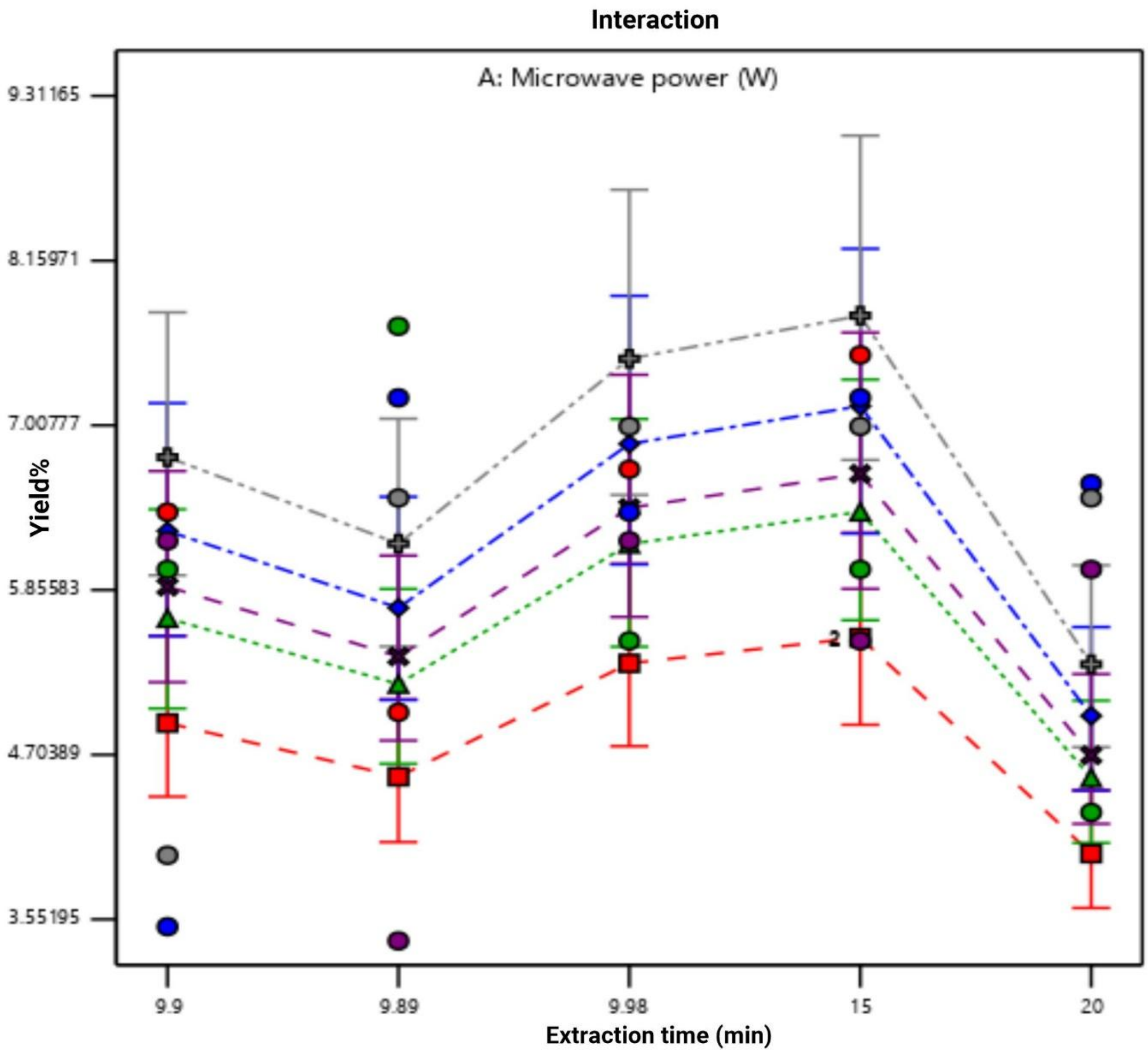


Fig 2. Demonstrates the interactions between factors such as extraction time (min), solvent volume (mL), and microwave power (W) using the 'One-Factor-At-Time' method.

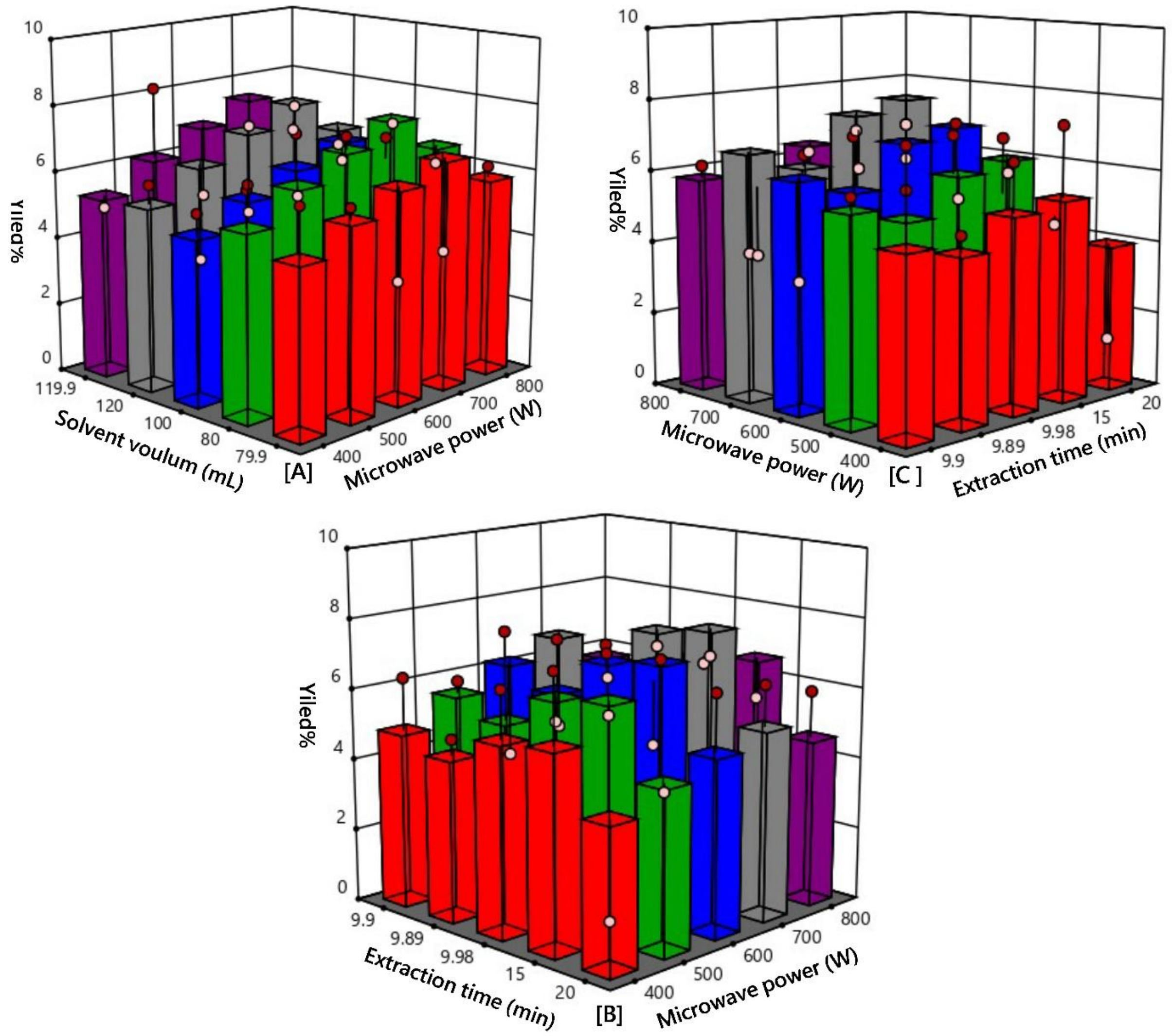
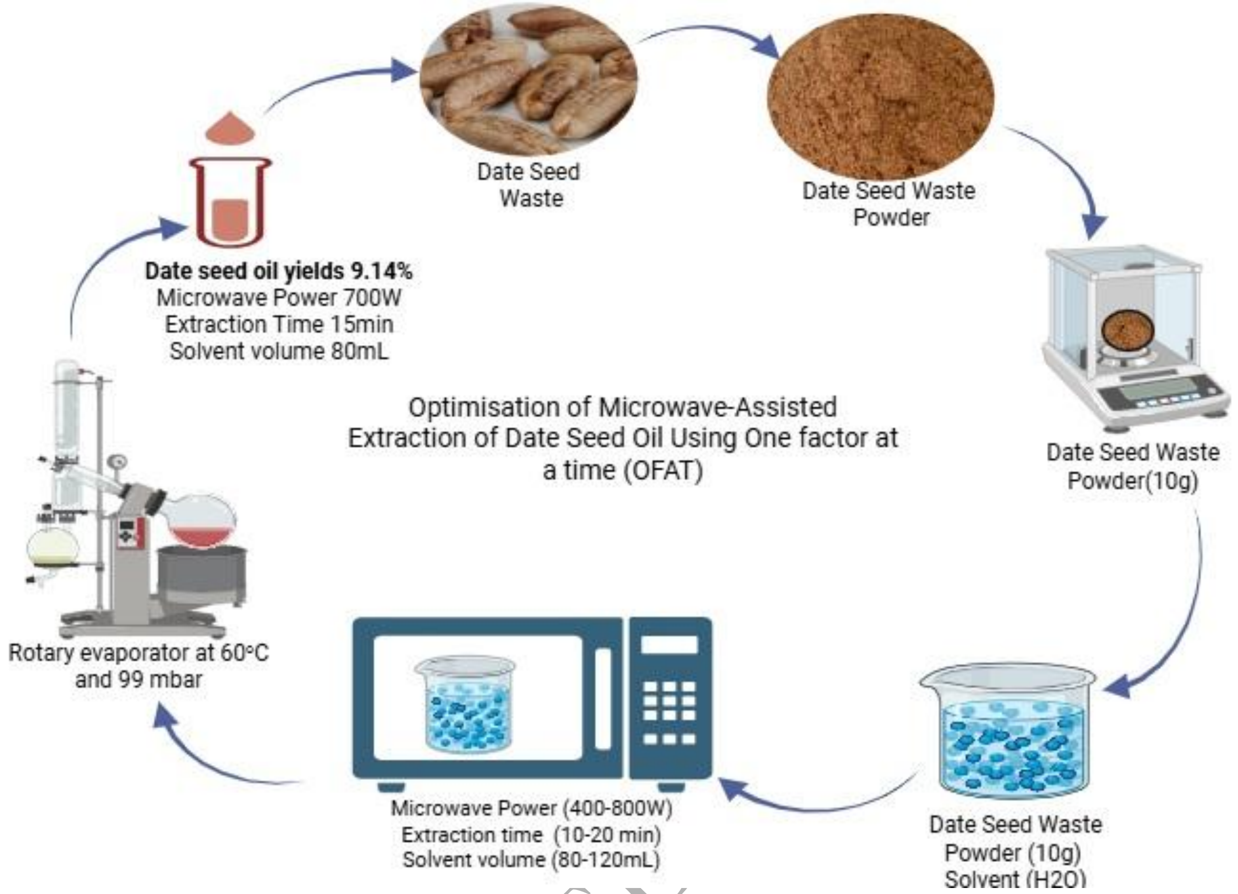


Fig 3.3D Response surface plots showing the effect of operating parameters on the yield % of seed oil extracted from date waste under different conditions. (A) Yield vs. microwave power at 700 W and solvent volume of 80 mL at a fixed extraction time (15 min); (B) yield vs. extraction time at (15 min) and microwave power of 700 W and fixed solvent volume of 80 mL; (C) yield vs. microwave power at 700 W and extraction time of (15 min) at field solvent volume of 80 mL.



Graphical abstract

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Table 1. Yield % of seed oil extracted from date waste under different conditions of MAE based on MCD Experiment (CDE) for response On-factor- at- time analysis

	Extraction condition			Analytical results
Run	X1: Microwave power (W)	X2: Solvent volume (mL)	X3: Extraction time (min)	Yield (%)
1	800	100	9.89	6.2
2	400	100	9.89	1.3
3	500	120	9.9	3.4
4	800	79.9	9.98	6.2
5	600	119.9	9.98	7.2
6	400	119.9	15	6.2
7	600	120	15	7.2
8	800	79.9	9.9	6.2
9	600	100	9.89	5.2
10	400	100	15	7
11	700	120	9.89	9.5
12	400	119.9	9.9	5

13	500	79.9	20	4.3
14	800	80	15	6
15	800	119.9	9.89	7.2
16	600	80	9.98	5.9
17	500	119.9	9.98	5.2
18	500	100	9.9	6
19	400	100	9.9	5.5
20	600	120	9.89	6.9
21	400	79.9	15	7.5
22	700	120	20	9
23	800	120	9.89	6
24	800	119.9	15	6.2
25	400	119.9	9.89	5.2
26	700	80	15	7.8
27	500	100	9.89	8.3
28	400	120	9.98	5.2
29	600	80	15	7.2
30	700	79.9	9.9	4
31	600	100	9.9	7.2
32	700	100	20	4.7
33	700	120	9.98	8
34	700	120	9.9	7.5
35	500	120	20	5.4
36	600	120	20	6.8
37	800	100	15	5
38	400	120	9.9	6
39	800	120	9.9	6
40	400	80	9.98	6.6
41	500	80	15	6.5
42	700	100	9.9	6.1
43	500	119.9	9.89	5.2
44	600	100	15	7
45	600	80	20	6.9
46	800	80	9.98	6.2
47	500	119.9	15	6.9
48	500	100	9.98	6
49	800	80	9.9	6
50	800	120	15	6.9
51	600	100	20	4.7
52	500	80	9.9	6
53	700	120	15	9.5
54	800	80	9.89	6
55	500	100	15	5
56	600	79.9	9.89	7.2
57	400	119.9	20	5
58	500	120	9.89	5.2
59	800	79.9	20	6
60	600	119.9	15	5.3

61	400	79.9	9.89	5
62	800	79.9	9.89	3.4
63	500	120	15	5.2
64	500	79.9	15	6
65	400	80	15	7
66	500	80	20	4.5
67	700	100	15	7
68	800	119.9	9.98	6.2
69	700	119.9	15	9.5
70	800	120	20	5
71	400	100	20	4.7
72	800	119.9	20	3.5
73	700	119.9	20	5
74	400	120	15	4.5
75	700	119.9	9.89	6.6
76	400	80	9.9	6.5
77	700	80	20	4
78	500	120	9.98	4.5
79	700	79.9	9.98	7
80	500	80	9.89	5.5
81	700	100	9.89	5
82	700	80	9.98	5.4
83	500	79.9	9.9	6
84	700	79.9	9.89	6.5
85	400	120	20	4.6
86	800	100	20	4.2
87	800	80	20	5
88	400	119.9	9.98	6
89	600	119.9	9.9	4.7
90	700	79.9	15	7
91	700	119.9	9.9	6.6
92	600	79.9	9.98	6.4
93	700	80	9.89	5.6
94	400	79.9	9.98	6.7
95	600	80	9.89	5.7
96	600	119.9	20	5.2
97	400	79.9	9.9	6.4
98	700	119.9	9.98	7.5
99	600	120	9.98	7
100	700	79.9	20	6.5
101	600	79.9	15	7.2
102	400	120	9.89	5.2
103	400	100	9.98	8.3
104	500	79.9	9.89	7.7
105	800	119.9	9.9	6.2
106	600	79.9	20	6.6
107	800	100	9.9	6.5

108	400	80	20	4.7
109	400	80	9.89	4
110	700	100	9.98	6
111	600	120	9.9	4.5
112	500	119.9	20	4.7
113	600	119.9	9.89	5.2
114	700	80	9.9	7.5
115	500	80	9.98	6.6
116	400	79.9	20	1.3
117	800	120	9.98	5.2
118	500	100	20	3.4
119	600	80	9.9	7.4
120	600	100	9.98	6
121	600	79.9	9.9	3.5
122	800	79.9	15	5.5
123	500	79.9	9.98	5.5
124	500	119.9	9.9	8.3
125	800	100	9.98	8.5

Table 2. ANOVA for MCD experiment (CDE) for response on-factor at-time analysis

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	**Significance**
Model	10.39	124	0.0838	4.66	<0.0001**	Significant
A-Microwave power	0.9764	4	0.2441	13.56	<0.0001**	
B-Solvent Volume	0.1559	4	0.039	2.17	0.0823	
C-Extraction time	1.51	4	0.3773	20.96	<0.0001**	
AB	0.9335	16	0.0583	3.24	<0.0001**	
AC	1.37	16	0.0855	4.75	<0.0001**	
BC	0.9541	16	0.0596	3.31	<0.0001**	
ABC	4.5	64	0.0702	3.9	<0.0001**	
Residual	1.154	64	0.018			
Lack-of-Fit	0.914	44	0.0208	1.73	>0.05	Not Significant
Pure Error	0.24	20	0.012			
Cor Total	11.544	188				

*CV=6.7%), PRESS=10.03
*P≤0.05 indicates that the model terms are significant.
**P≤0.05 indicates the model terms are highly significant.

Table 3 shows the optimal conditions for validating the date seed oil extraction

Dates seed oil extraction	**Values**	
	predicted	Experimental (n=3)

Yield %	6.2448	9.14±0.124

All reported values are presented as mean ± standard deviation. Within each row, values that do not share a common superscript letter (e.g., 'a') are considered statistically significant at $p \leq 0.05$.

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