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Optimization of process variables on physicochemical properties of milk during an innovative refractance window concentration

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Abstract

An innovative refractance window (RW) concentrator was developed and used to concentrate milk samples at temperatures of 50–70°C and pressures of 0.4–0.8 bar. Optimum process conditions were found through response surface methodology to compare RW at optimal conditions with the conventional concentration (CC). Also, the effects of process parameters and their interactions on the RW process and product specifications were analyzed. According to the results, optimal RW conditions were a pressure of 0.4 bar and a temperature of 69.1°C. Besides, the energy efficiency and overall heat transfer coefficient of RW at optimal conditions were higher than those of CC by 125.2% and 15.3%, respectively. Compared to CC, RW reduced the concentration-time by 60.8% and minimized the changes in the chemical composition and color of the milk. RW process was found to be a time- and energy-saving concentration technique that can produce concentrated milk with improved quality.

Practical applications

Alternative concentration techniques that can reduce energy consumption and processing time while guaranteeing the quality retention of the product might be considered by the food industry. The innovative RW milk concentration method that was developed in the present study showed to be a promising time- and energy- saving concentration technique. This method was able to retain the quality characteristics of milk during the concentration process. Also, the optimization information provided in the present study can be used for developing upscaled units that can be used for the commercial concentration process of milk.

1 | INTRODUCTION

Cow milk is a nutritionally valuable product containing many of the essential macro and micronutrients including proteins, carbohydrates, fats, minerals, vitamins, and bioactive components (Al-HilphyShirkole, Ali, & Mohsin, 2019; Stratakos et al., 2019). Milk produced in the farm is usually processed to enhance its safety and shelf-life or to produce several dairy products (Musina et al., 2018; Stratakos et al., 2019). For example, the shelf-life of the milk can be enhanced by producing condensed milk which involves evaporative operations (Guimarães, Martins, Flauzino, Basso, & Telis Romero, 2020). Also, milk and whey protein powders can be produced through the evaporation process to be used in dairy product formulation (Musina et al., 2018). In either case, retaining the quality parameters, such as product color, is among the technical considerations in the concentration unit operation as it can affect the acceptance of the product (Faion, Becker, Fernandes, Steffens, & Valduga, 2019; Fernández-Vázquez et al., 2018).

As the traditional concentration methods may negatively affect the physicochemical properties of milk, researchers are exploring the applicability of emerging technologies for milk concentration to produce a high-quality product (Faion et al., 2019; Moejes, vanWonderen, Bitter, & vanBoxtel, 2020; Parmar, Singh, Meena, Borad, & Raju, 2018). The refractance window (RW) is a novel food processing technique that is believed to produce high-quality products and has been used for drying food material at both laboratory and industrial scales (Bernaert, VanDroogenbroeck, VanPamel, & DeRuyck, 2019; Jafari, Azizi, Mirzaei, & Dehnad, 2016; Raghavi, Moses, & Anandharamakrishnan, 2018). However, there is limited information about the applicability of this technology for the concentration of food materials such as milk. Hence, the present study aims to investigate the potential use of RW technology for milk concentration. Moreover, it focused on understanding the effects of RW process parameters, including process temperature and pressure, on the performance of RW milk concentrator and chemical composition of the product, as well as the overall color change of the product. The suitability of the proposed data modeling approach was also verified by comparing the predicted data with those of experimental analysis.

2 | MATERIALS AND METHODS

2.1 | Raw materials

Fresh cow milk was obtained from the Agricultural Researches Station of the University of Basrah and was stored at 5°C. Before concentration processes, the milk temperature was increased from 5 to 40°C using a laboratory water bath (GFL 1008, German) and then the milk was standardized by the adding milk powder (Alsabah, Iran) and dairy cream (Barmezan, Iran) to adjust the TSS and fat content of the raw materials using Pearson square (Figure 1). The standardized milk samples were then concentrated using either RW or conventional concentrator.

2.2 | Milk concentration processes

An RW system, designed and developed at the Department of Food Science of the University of Basrah, was used to concentrate milk. This device consists of a concentration unit made of Pyrex glass (with the inner diameter of 6 cm, the height of 84 cm, and a thickness of 0.2 cm), a 2 kW heating unit (which heats the water as a heat transfer medium for circulation), and a control unit. A schematic representation of the RW system was prepared using Actrix Technical 2000 Software (Autodesk Inc., USA) (Figure 2). For the RW concentration process, 5L of the standardized milk sample was placed in the RW system and heated to 95°C. Afterward, partial vacuums (0.4–0.8 bars, depending on the treatment) were applied, corresponding to the milk boiling point of 50–70°C. The sample was held at these conditions to the time that the desired concentration, i.e., 26% TSS, was achieved.

Besides, 5 L of milk samples were conventionally concentrated (CC) to 26%TSS using an electric heater (Orbon, India) at a power of 2 kW. All the concentration processes were repeated for three times. Besides, the temperature of the milk during the process was monitored using a digital temperature controller (LTR5/LEA Electronic, Italy).

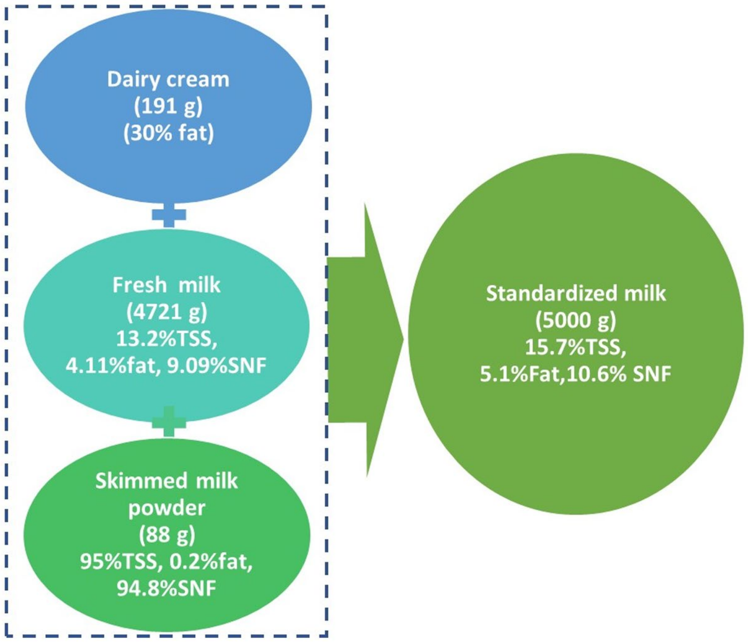

FIGURE 1 The standardization process of milk in the present study. SNF, solid non-fat; TSS, total soluble solid

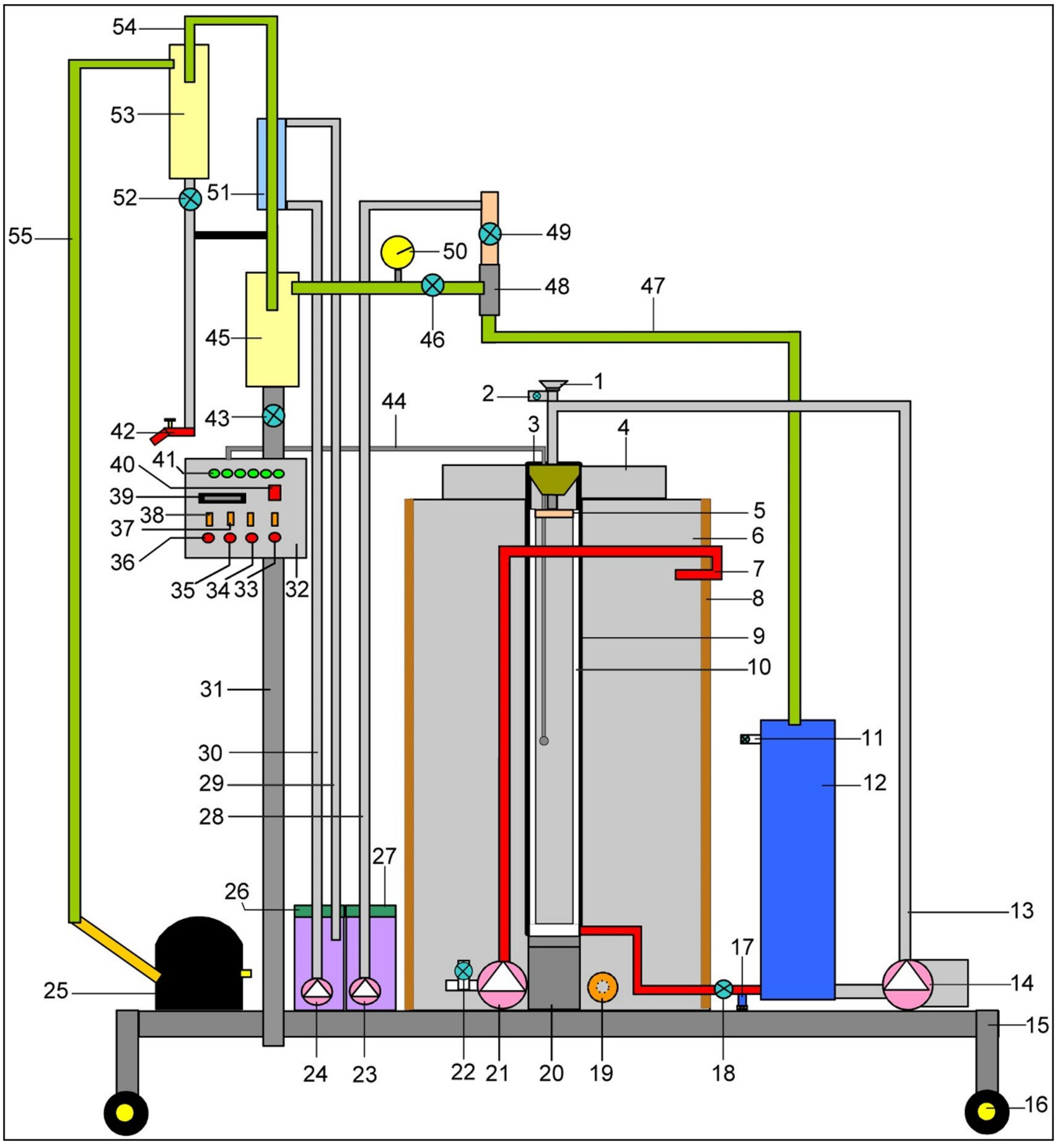

FIGURE 2 Schematic representation of the refractance window (RW) system used in the present study ((1) The milk inlet hole, (2) Milk inlet valve, (3) Plastic cap, (4) Tank cap, (5) Milk distribution tube, (6) Water tank, (7) Hot water circulation tube, (8) Insulator, (9) Glass tube (Pyrex), (10) Milk layer inside tube, (11) Vent valve, (12) Plastic cylinder, (13) Milk transfer tube, (14) Centrifugal pump, (15) Iron base, (16) Tire, (17) Electric valve, (18) Manual valve, (19) Electric heater, (20) Iron centrifuge pump, (22). Manual valve, 24&23. Plunger, (25) A vacuum pump, 27&26. Plastic tanks, (28). Washing water circulation tube, (29). The return water pipe from the heat exchanger, (30). Cold water transfer tube to the heat exchanger, (31). Iron shaft, (32). Control panel, (33). Water circulation pump switch, (34). Washing pump operation switch, (35) Milk Recycle Pump Operation switch, (36). Heat exchanger water circulation operation switch, (37). Vacuum pump operation switch, (38). Milk exit electric valve operation, (39). Digital temperature gauge, (40). Heater operation switch, (41). Signal lamps, (42). Tap for condensed water out, (43). Valve, (44). Thermocouple for temperature measurement, (45). Steam trap, (46). Valve, (47). Discharge tube, (48). Partition, (49). Valve, (50). Vacuum pressure gauge, (51). Heat exchanger, (52). Valve, (53). Steam trap, (54). Tube, (55). Vacuum tube, (56) Valve)

2.3 | Process evaluation

2.3.1 | Specific energy consumption and energy efficiency

Specific energy consumption (SEC) and energy efficiency (η) were calculated to analyses the energy consumption for RW and CC milk concentration (Equations 1-2) (Chamberland et al., 2020).

$$SEC\,(\mathrm{kJ/kg}) = \frac{Q_{in}}{M_w} \tag{1}$$

$$\eta = \frac{Q_0}{Q_{in}} \times 100 \tag{2}$$

where, Q_{in}, Q_o and M_w are input, output energy (kJ) and mass of evaporated water (kg).

2.3.2 | Overall heat transfer coefficient

Overall heat transfer coefficient was calculated using Equation (3) (Pehlivan & Özdemir, 2012).

$$U = \frac{MC_P\left(T_{h0} - T_{m0}\right) + m_w \lambda_w}{A\ \Delta T_m} \tag{3}$$

where, U is the overall heat transfer coefficient (W/m^2°C), A is the area (m^2), ΔT_m is the logarithmic mean temperature difference (LMTD). M is the mass flow rate of milk (kg/s), C_p is the specific heat (kJ/kg°C), (T_{h0}–T_{m0}) is the temperature difference between the temperature of hot water (°C) and temperature of hot milk (°C) respectively,m_w mass of evaporated water from milk (kg/s) and λw is the latent heat (kJ/kg). ΔT_m can be calculated according to Equation (4) as follows:

$$\Delta T_m = \frac{\left(T_{hi} - T_{mi}\right) - \left(T_{h0} - T_{m0}\right)}{\ln\left[\frac{T_{hi} - T_{mi}}{T_{h0} - T_{m0}}\right]} \tag{4}$$

where, (T_{hi}–T_{mi}) is the difference between cold water temperature (°C), and cold milk temperature (°C) respectively.

2.4 | Chemical analyses

All the chemical assays in the present study, including determination of moisture content, protein, fat, pH, ash, lactose and TSS, were performed according to the Official Methods of Analysis (AOAC, 2016).

2.5 | Color measurement using computer vision

A 720p HD camera (IP67 Endoscope, Mileseey, China), and four light-emitting diodes (LED) lamps (LB13W, Konnice Co., China) that are located on the top of a wooden-black color box. Two lamps have a 45° angle with the camera lens and two are perpendicular to the sample. The image processing technique was employed to analyze the color characteristics of milk samples in triplicate similar according to that described in the literature with some modifications (Gavahian, Farahnaky, Javidnia, & Majzoobi, 2012; Yam & Papadakis, 2004). In this regard, the digital images obtained and the ImageJ software (Version 1.52q, National Institutes of Health, United States) was utilized to obtain CIE $L^*a^*b^*$ values from digital images. These parameters were used to calculate overall color changes (ΔE). This parameter represents the effects of concentration processes on the sample color, that is, a higher ΔE value indicates more changes in the milk color after the concentration process. In this regard, the color values of the concentrated milk samples were compared to those of raw milk (Equation 5) (Gavahian, Sheu, Tsai, & Chu, 2020; Wasnik et al., 2019).

$$\Delta E = \sqrt{\left(L_o^* - L^*\right)^2 + \left(a_o^* - a^*\right)^2 + \left(b_o^* - b^*\right)^2} \tag{5}$$

where ΔE is overall color change, $L_o{}^*$, $a_o{}^*$, and$b_o{}^*$are lightness, redness-greenness, and yellowness-blueness of raw milk (before the concentration process) and L^*, a^*, and b^* are those of the concentrated samples, respectively.

2.6 | Experimental design

The preliminary trials were carried out to select the range of independent variables, i.e., pressure (0.4, 0.6, and 0.8 bar) and temperature (50, 60, and 70°C). Furthermore, the central composite design (CCD) (3 × 3) of response surface methodology (RSM) (Design-Expert Software, version 7, Stat-Ease Inc., United States) was employed to assess the effects of independent variables and their interactions on the U, concentration-time (CT), SEC, η, moisture content, ash pH, fat, protein, lactose, TSS, ΔE. In this regards, thirteen experiments were used in CCD for each of the pressure values with five replications at the center point with two axial points. The independent variables ranges are presented in Table 1. The codes in the CCD were −1, 0, +1 epitomized the lowest, medium, and highest values, respectively.

2.7 | Process optimization and data modeling

Graphical and numerical methods were applied to optimize milk concentration conditions (independent variables), i.e., pressure (0.4–0.8 bar) and the temperature (50–70°C) of the holding phase, to obtain a milk concentrate with good thermal performance and chemical properties. The minimum values of CT, moisture content, SEC, and ΔE as well as the maximum values of U, energy efficiency, ash, pH, fat, protein, lactose, and TSS were targeted in the optimization process using version 7 of Design-Expert software (Stat-Ease Inc., United States) at various conditions of milk concentration process (0.4-0.8 bar pressure, 50–70°C temperature). After that, functions of the desirability were developed for the dependent variables (U, CT, SEC, η, moisture content, ash, pH, fat, protein, lactose, TSS, and ΔE). Besides, the optimum combinations of pressure and temperature were selected via response surface plots of the dependent variables, and the desirability function method was used to optimize the multiple responses according to Equation 6. In the present study, the desirability (D) values range between 0 and 1 and the impotence term (r_i) ranges between 1–5 considered as appropriate values for the optimization purposes (Eren & Kaymak-Ertekin, 2007).

$$D(x) = \left(d_1^{r_i} \times d_2^{r_i} \times d_3^{r_i} \times \ldots\ldots\ldots\ldots \times d_n^{r_i}\right)^{1/\sum r_i} = \left(\prod_{i=1}^{n} d_i^{r_i}\right)^{1/\sum r_i} \tag{6}$$

where d_i is the desirability for each response, r_i is a number refers to the relative importance of the i response, where 5 is the greatest importance and 1 is the minimum importance.

To predict the dependent variables, the quadratic polynomial regression model was used as given in Equation 7 (Khuri & Cornell, 2019).

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TABLE 1 Central composite design matrix for the effect of pressure and temperature on the dependent variables of the milk concentrated by the refractance window along with p-value, R^2 and Lack of Fit of model.

RW run	P (bar)	T (°C)	U (W/m^2.°C)	CT (h)	SEC (kJ/kg)	η (%)	MC (%)	Pr. (%)	Ash (%)	Fat (%)	Lact. (%)	pH	TSS (%)	ΔE
1	0.4	50	70.140 ± 0.14^{h}	1.12 ± 0.01^{c}	2,752.49^{c} ± 6.11	62.20^{c} ± 1.09	74.00^{c} ± 0.32	7.72^{b} ± 0.02	1.20^{c} ± 0.01	7.50^{b} ± 0.12	9.58^{c} ± 0.12	6.50^{b} ± 0.02	26.00^{a} ± 0.22	10.77^{c} ± 0.12
2	0.8	50	168.33 ± 1.91^{c}	3.30 ± 0.02^{a}	6,605.00^{a} ± 5.34	34.30^{j} ± 0.98	76.01^{a} ± 0.93	7.10^{b} ± 0.02	1.10^{e} ± 0.2	7.30^{d} ± 0.03	8.45^{f} ± 0.23	6.40^{c} ± 0.01	24.00^{c} ± 0.76	7.63^{d} ± 0.23
3	0.8	60	217.56 ± 2.43^{b}	3.22 ± 0.03^{a}	6,606.01^{a} ± 3.89	43.72^{h} ± 1.04	76.00^{a} ± 0.21	7.45^{b} ± 0.01	1.00^{d} ± 0.01	7.30^{d} ± 0.06	8.25^{g} ± 0.42	6.51^{b} ± 0.02	24.00^{c} ± 0.98	11.52^{b} ± 0.11
4	0.8	70	252.49 ± 2.71^{a}	3.00 ± 0.07^{a}	6,607.03^{a} ± 8.67	49.68^{e} ± 2.10	75.00^{b} ± 0.24	7.32^{b} ± 0.02	1.08^{d} ± 0.03	7.40^{c} ± 0.07	9.20 ± 0.45	6.60^{a} ± 0.04	25.01^{b} ± 0.03	9.52^{c} ± 0.22
5	0.6	60	142.00 ± 0.98^{d}	2.00 ± 0.06^{b}	4,403.98^{b} ± 9.65	46.65^{g} ± 0.89	74.02^{c} ± 0.16	7.13^{b} ± 0.03	1.42^{a} ± 0.02	7.66^{a} ± 0.05	9.79^{a} ± 0.11	6.60^{a} ± 0.02	26.00^{a} ± 0.97	11.46^{b} ± 0.21
6	0.4	70	105.20 ± 1.30^{e}	1.00 ± 0.01^{d}	2,752.50^{c} ± 5.99	71.80^{a} ± 0.56	74.01^{c} ± 0.52	8.12^{a} ± 0.02	1.20^{c} ± 0.04	7.30^{d} ± 0.08	9.38^{d} ± 0.23	6.54^{b} ± 0.02	26.01^{a} ± 0.93	12.48^{a} ± 0.22
7	0.4	60	90.650 ± 1.60^{g}	1.10 ± 0.02^{c}	2,752.50^{c} ± 6.98	66.88^{b} ± 0.96	74.00^{c} ± 0.43	7.51^{b} ± 0.04	1.43^{a} ± 0.03	7.40^{c} ± 0.04	9.66^{c} ± 0.09	6.50^{b} ± 0.01	26.00^{a} ± 0.44	9.97^{c} ± 0.34
8	0.6	60	145.04 ± 2.02^{d}	2 ± .010.09^{b}	4,403.99^{b} ± 9.11	47.44^{f} ± 1.20	74.02^{c} ± 0.82	7.27^{b} ± 0.03	1.38^{b} ± 0.04	7.50^{b} ± 0.07	9.85^{a} ± 0.14	6.53^{b} ± 0.04	26.00^{a} ± 0.06	11.45^{b} ± 0.33
9	0.6	60	145.04 ± 2.11^{d}	2.05 ± 0.08^{b}	4,404.0^{b} ± 11.10	48.85^{e} ± 0.99	75.01^{b} ± 0.47	6.96^{c} ± 0.11	1.24^{c} ± 0.05	7.40^{c} ± 0.05	9.40^{d} ± 0.15	6.41^{c} ± 0.03	25.02^{b} ± 0.04	10.50^{c} ± 0.22
10	0.6	60	145.04 ± 2.09^{d}	2.00 ± 0.07^{b}	4,403.60^{b} ± 12.34	48.85^{e} ± 1.09	75.00^{b} ± 0.17	7.80^{b} ± 0.14	1.00^{d} ± 0.04	7.30^{d} ± 0.09	8.90^{e} ± 0.22	6.50^{b} ± 0.01	25.00^{b} ± 0.11	11.46^{b} ± 0.13
11	0.6	70	168.33 ± 1.89^{c}	1.98 ± 0.02^{b}	4,403.90^{b} ± 8.43	55.24^{d} ± 2.03	75.00^{b} ± 0.11	6.95^{c} ± 0.06	1.25^{c} ± 0.01	7.50^{b} ± 0.10	9.30^{d} ± 0.34	6.51^{b} ± 0.05	25.00^{b} ± 0.01	11.12^{b} ± 0.43
12	0.6	50	112.22 ± 2.30^{e}	2.10 ± 0.01^{b}	4,404.01^{b} ± 5.67	39.64^{i} ± 0.96	74.01^{c} ± 0.0.05	7.21^{b} ± 0.05	1.42^{a} ± 0.03	7.68^{a} ± 0.08	9.69^{c} ± 0.33	6.60^{a} ± 0.02	26.00^{a} ± 0.83	11.51^{b} ± 0.45
13	0.6	60	145.04 ± 2.55^{d}	2.04 ± 0.02^{b}	4,404.30^{b} ± 6.13	48.85^{e} ± 3.01	75.00^{b} ± 1.24	7.35^{b} ± 0.01	1.25^{c} ± 0.02	7.40^{c} ± 0.04	9.00^{e} ± 0.13	6.63^{a} ± 0.04	25.00^{b} ± 1.01	11.45^{b} ± 0.56
p-value of model			<.0001	<.0001	.0001	<.0001	.0188	.3394	.4868	.2415	.1446	.8127	.0084	.009
R^2			0.9994	0.9986	0.9787	0.9936	0.6155	0.4946	0.4138	0.5557	0.6296	0.2364	0.6525	0.9376
LoF			0.2726	0.110	0.1246	0.3701	0.6270	0.4613	0.5946	0.8415	0.6489	0.6126	0.6270	0.083

The different l letters in the columns indicate a significant effect at level of 0.05.

Abbreviations: CT, concentration-time; LoF, lack of fit; MC, moisture content; Pr, protein; P, pressure; Lact., lactose; SEC, specific energy consumption; SS, sum of square; T, temperature; TSS, total soluble solids; U, heat transfer coeffecient; η, energy effeciency; ΔE, total color change.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{i<j=1}^{k} \beta_{ij} X_i X_j \qquad (7)$$

where Y is the responses (U, CT, SEC, η, moisture content, ash pH, fat, protein, lactose, TSS, and ΔE), X is the independent variables, k is the number of factors, i and j are factors numbers, β_0 is a constant, and β_i, β_{ii} and β_{ij} are coefficients of linear, quadratic, and interaction terms, respectively.

2.8 | Statistical analysis

Statistical analysis was carried out using Design-Expert software (version 7, Stat-Ease Inc., United States). Analysis of Variance (ANOVA) was exploited to evaluate the differences between the means. All components in the polynomial models were evaluated statistically using p-value at .05 level. The results in the current study were represented as means ± standard deviations and all experiments were carried out in triplicates.

3 | RESULTS AND DISCUSSION

3.1 | Performance of RW concentrator

3.1.1 | Overall heat transfer coefficient

The results showed that the U values ranged between 70.140 W/m^2°C at a pressure of 0.40 bar and a temperature of 50°C to 252.49 W/m^2°C at a pressure of 0.80 bar and a temperature of 70°C (Table 1). This may be due to an increase in temperature. Jebson and Iyer (1991) found that the U values lie between 0.3–3.2 kW/m^2°C for concentrated skim milk in a 5-stage multi-effect evaporator. On the other hand, they mentioned that the values of U ranged between 0.8–3.08 kW/m^2°C for whole milk (Jebson & Iyer, 1991). In another study, the total heat transfer coefficient of milk ranged from 477 to 939 W/m^2°C when the scraper thin film evaporator was used (Sangrame, Bhagavathi, Thakare, Ali, & Das, 2000). Silveira et al. (2013) stated that the total heat transfer coefficient for the concentration of skimmed milk under vacuum ranged between 1.24–1.93 kWh/m^2°C in the first stage and ranged between 1.25–2.00 kW/m^2°C in the second stage. They emphasized that the heat transfer coefficient was not significantly affected by the conditions of concentration (Silveira et al., 2013).

The results of the statistical analysis in Table 1 showed that there was a significant effect (p < .05) of the mathematical model. Also, the Lack of Fit (LoF) was not significant (p > .05). It has been noted that R-Squared was 0.9994. These indicators showed that the multiple nonlinear correlation equation can be used to predict U values. In addition, the regression coefficients of models are presented in Table 2. Furthermore, Equation (8) is used to calculate U as a function of temperature and pressure.

$$U = -78.405 - 334.957P + 4.756T - 6.1369PT + 230.887P^2 - 0.045966T^2 \qquad (8)$$

TABLE 2 Regression coefficient for response surface linear, quadratic and reduced cubic models for dependent variables

Source	U (W/m^2 °C)	CT (h)	SEC (kJ/kg)	η (%)	MC (%)	Pr. (%)	Ash (%)	Fat (%)	Lact. (%)	pH	TSS (%)	ΔE
Intercept	-78.405	-1.038	1,107.952	107.342	67.692	7.108	0.894	10.792	16.771	7.268	32.307	196.831
P	-334.957	3.486	1,360.222	−317.663	11.666	−9.669	3.027	2.279	2.287	−0.123	−11.666	−225.516
T	4.756	0.023	−0.104	1.1367	0.075	0.108	-8.90×10^{-3}	−0.127	−0.249	−0.025	−0.075	−7.305
P × T	−6.136	−0.022	0.249	0.722	−0.12500	−0.022	2.500×10^{-3}	−0.03750	−0.118	−0.020	0.125	11.1426
P^2	230.887	2.594	6,882.153	177.797	-	8.155	−2.84914	−3.91379	−9.732	−0.9181	-	−154.859
T^2	−0.045	-1.62×10^{-4}	-1.14×10^{-4}	-7.44×10^{-3}	-	-7.37×10^{-4}	6.03×10^{-5}	8.34×10^{-4}	1.5061	1.32×10^{-4}	-	0.067

Abbreviations: CT, concentration-time; Lact., lactose; MC, moisture content; Pr., protein; P, pressure; SEC, specific energy consumption; SS, sum of square; T, temperature; TSS, total soluble solids; U, heat transfer coeffecient; η, energy effeciency; ΔE, total color change. P^{2}T and PT^2 for ΔE were calculated as +2.229 and -0.114, respectively.

Concerning the interference between pressure and temperature, Figure 3a, which was drawn using the response surface methodology, the value of the overall heat transfer coefficient was 70.18 W/m^{2}°C when using a pressure of 0.40 bar and a temperature of 50°C. it was observed that it increased to 196 W/m^{2}°C when using a pressure of 0.80 bar and a temperature of 50°C. This is because the increase of pressure led to a decrease in evaporation and thus the temperature remains more stable. It was also observed that U rose from 104 W/m^{2}°C to 252 W/m^{2}°C when using a temperature of 70°C and a pressure of 0.80 bar. This is due to the high temperature, which increases the overall heat transfer coefficient. As shown in Figure 3a, the interference between pressure and temperature can increase the overall heat transfer coefficient better, especially when using their maximum values.

3.1.2 | Concentration-time

It is observed from Table 1 the central composite design matrix for the effect of pressure (bar) and temperature (°C) on the CT of the RW process. The results showed that the concentration of milk by the RW milk concentrator required less time which reached 1 hr when using a pressure of 0.40 bar and a temperature of 70°C. The longest CT was 3.3 hr at a pressure of 0.80 bar and a temperature of 50°C. The results indicated that the effect of pressure was greater than the effect of temperature, as pressure was the main factor affecting the CT.

The statistical analysis (Table 1) showed a significant effect ($p < .05$) for the mathematical model. In addition, LoF was not significant ($p > .05$). It has been noted that R-Squared was 0.9986. These indicators showed that the mathematical model applies to data and can be used to predict the CT and the coefficient of models are depicted in Table 2 and Equation(9) which used to calculate CT as follows:

$$CT = -1.03897 + 3.48621P + 0.023948T - 0.022500PT + 2.59483P^2 - 1.6206910^{-4}T^2 \quad (9)$$

Regarding the interference between pressure and temperature, the three-dimensional Figure 3b that was drawn by the response surface methodology. The CT value was 1.11 hr when using pressure

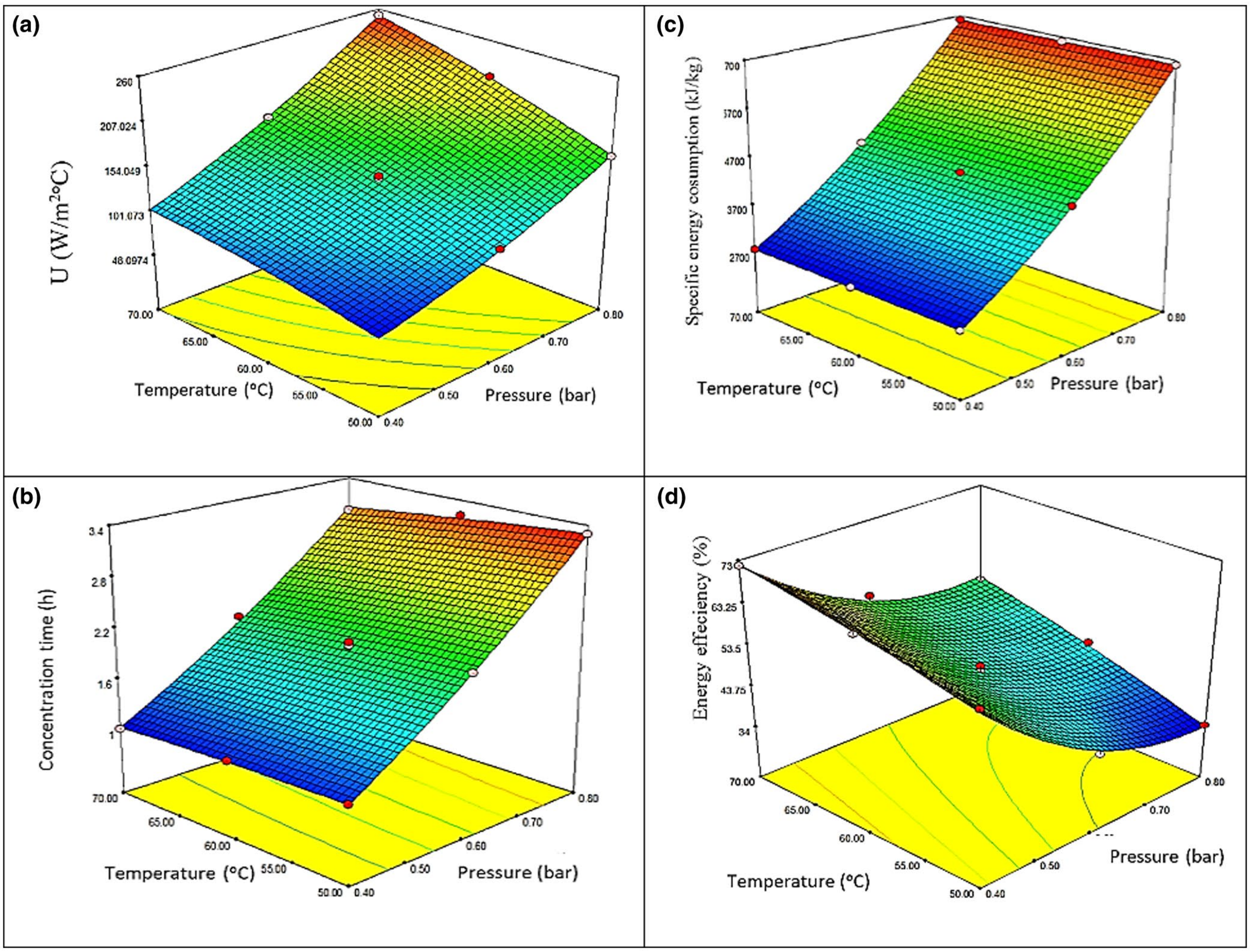

FIGURE 3 Response surface plot of (a) heat transfer coefficient, (b) concentration-time (c) specific energy consumption, (d) energy efficiency as a function of process temperature and pressure

0.40 bar and temperature 50°C, and the CT increased to 3.30 hr when using pressure 0.80 bar and a temperature of 50°C. In general, lowering the pressure and increasing the temperature can reduce the CT due to withdrawal of the largest amount of moisture per unit of time and rapid evaporation, respectively (Silveira et al., 2013; Yanniotis, 2007).

Specific energy consumption

The results showed that the values of the SEC ranged between 2,752.49 kJ/kg at a pressure of 0.4 bar and temperature of 50°C to 6,607 kJ/kg at a pressure of 0.80 bar and a temperature of 50°C (Table 1). Silveira et al. (2013) found that the specific energy consumption of the skim milk concentration was 3,024 kJ/kg vaporized water in the first stage, and 2,889 kJ/kg evaporated water in the second stage in the two-stage evaporator. Yanniotis (2007) showed that multi-effect evaporators reuse the latent heat of the steam and thus save energy.

The statistical analysis in Table 1 showed a significant effect ($p < .05$) of the mathematical model. Besides, the LoF was not significant ($p > .05$). Besides, high *R*-Squared values indicated that the mathematical model well-predicted the values of U, SEC, CT, and η. Moreover, according to the coefficients of models in Table 2, and Equation (10) was proposed to calculate the SEC.

$$SEC = 1107.9523 + 1360.2225\mathrm{P} - 0.1041\mathrm{T} + 0.2492\mathrm{PT} + 6882.1534P^2 - 1.1473 \times 10^4 T^2 \tag{10}$$

Regarding the interference between pressure and temperature, the three-dimensional Figure 3c that was drawn by the response surface methodology. The value of SEC was 2,752.5 kJ/kg when using a pressure of 0.40 bar and a temperature of 70°C and decreased to 2,752.49 kJ/kg at a pressure of 0.40 bar and temperature of 50°C.

3.1.3 | Energy efficiency

The results showed that the η ranged between 34.30% at a pressure of 0.80 bar and a temperature of 50°C to 71.80% at a pressure of 0.40 bar and a temperature of 70°C as presented in Table 1. Silveira et al. (2013) found that η was 79% and 81% in the first and second stages of the concentration process for skim milk, respectively.

The results of the statistical analysis in Table 1 showed that there was a significant effect ($p < .05$) of the mathematical model in η. The results of the statistical analysis showed that the LoF was insignificant ($p > .05$). R^2 was 0.9936. These indicators showed the possibility of using the nonlinear mathematical model in predicting η values and the model coefficients are illustrated in Table 2, and Equation (11) that used to calculate η as follows:

$$\eta = 107.34270 - 317.66383\mathrm{P} + 1.13674\mathrm{T} + 0.72208\mathrm{PT} + 177.79707P^2 - 7.44567 \times 10^{-3} T^2 \tag{11}$$

Regarding the interference between pressure and temperature, the three-dimensional Figure 3d that was drawn by the response surface methodology. The η value was 61.38% when using a pressure of 0.40 bar and a temperature of 50°C and decreased to 34.10% at a pressure of 0.80 bar and a temperature of 50°C. η reached 72.03% when using a pressure of 0.40 bar and a temperature of 70°C and decreased to 50.52% at a pressure of 0.80 bar and a temperature of 70°C. This may be due to the decrease in pressure and the increase in the temperature led to an increase in η. Lowering the pressure and increasing the temperature were increased the amount of evaporated water and reducing CT (Silveira et al., 2015).

3.2 | Chemical composition

3.2.1 | Moisture content

According to Table 1, the values of moisture content ranged between 74% at a pressure of 0.40 bar and a temperature of 50°C to 76.01% at a pressure of 0.80 bar and a temperature of 50°C. Also, there was a significant effect ($p < .05$) of the mathematical model. Besides, the LoF was not significant ($p > .05$). A linear model was proposed to predict the moisture content (MC) of concentrated milk as a function of process temperature (T) and pressure (P) (Table 2 and Equation 12).

$$\mathrm{MC} = 67.69231 + 11.66667\mathrm{P} + 0.075000\mathrm{T} - 0.12500\mathrm{TP} \tag{12}$$

For the interference between pressure and temperature as shown in the three-dimensional Figure 4a that was drawn by the response surface methodology, the value of moisture content was 73.60% when using the pressure 0.40 bar and a temperature of 50°C and increased to 75.28% at a pressure of 0.80 bar and a temperature of 70°C. The moisture content increased from 74.10% at a pressure of 0.40 bar and a temperature of 70°C to 75.27% at 0.80 bar and a temperature of 70°C. This is because the increase in pressure leads to a decrease in the vacuum inside the cylinder, which leads to a reduction of the amount of water evaporated from the milk (Silveira et al., 2013; Tanguy et al., 2016). Yanniotis (2007) showed that concentration is used to reduce the weight and volume of liquid products as the concentration reduces the water activity in the nutrient.

3.2.2 | Protein

According to Table 1, the protein values ranged between 6.95% at a pressure of 0.60 bar and a temperature of 70°C to 8.12% at a pressure of 0.40 bar and a temperature of 70°C (Table 1). These results were close to the results of a research team that stated the percentage of protein in concentrated milk was 8.6% (Madoumier, Azzaro-Pantel, Tanguy, & Gésan-Guiziou, 2015).

The not significance of the quadratic model and the LoF on the protein content is presented in Table 1. The concentration process affected the physical and chemical conditions of the product. Also, the reaction of Maillard occurs during the concentration process, due

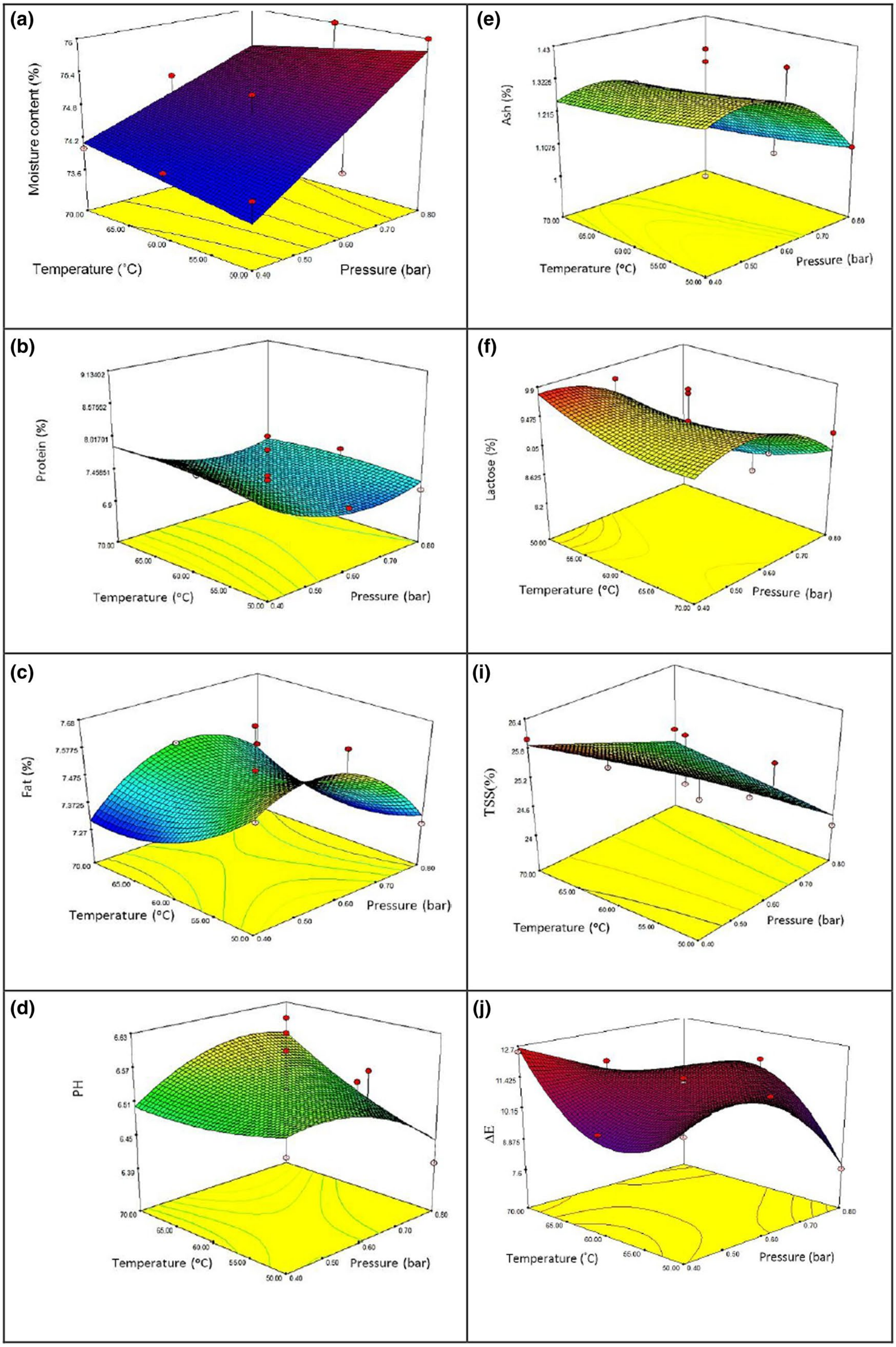

FIGURE 4 Response surface plot of (a) moisture content, (b) protein (c) fat, (d) pH, (e) ash (f) lactose, (i) TSS, (j) *ΔE* as a function of process temperature and pressure

to the increase in the amount of evaporated water due to the high temperature that led to the increase in protein (Tanguy et al., 2016). Table 2 shows the regression coefficients and Equation (13) was used to calculate the protein (Pr) of concentrated milk.

$$Pr = 7.10 - 0.9699\mathrm{P} + 0.108\mathrm{T} - 0.022\mathrm{PT} + 8.1551P^2 - 7.379 \times 10^{-4}T^2 \tag{13}$$

Regarding the interference between pressure and temperature, as shown in the three-dimensional Figure 4b that was drawn by the response surface methodology. To obtain the highest percentage of protein, the pressure of 0.40 bar and the temperature of 70 °C was used to reach 7.86%, then followed by the pressure of 0.40 bar and temperature 50°C, where it amounted to 7.65%. The increase in pressure causes a decrease in protein content at different temperatures. For example, the protein decreased insignificantly ($p > .05$) from 7.86% to 7.28% when the pressure increased from 0.4 to 0.8 bar and a temperature of 70°C.

3.2.3 | Fat

The results showed that the fat values ranged between 7.3% at pressure 0.80 bar and a temperature of 50°C to 7.68% at a pressure of 0.60 bar and a temperature of 50°C (Table 1). These results are closed to the findings of Madoumier el al. (2015) who indicated that the percentage of fat for concentrated milk was 7.0%. Also, Table 1 showed no significant effect ($p > .05$) for the quadratic model. For the interaction between pressure and temperature as shown in the three-dimensional Figure 4c, the fat content was 7.5% when using pressure 0.60 bar and a temperature of 50°C, and decreased insignificantly ($p > .05$) to 7.3% at a pressure of 0.80 bar and a temperature of 50°C. Equation(14) was used to calculate the fat content of concentrated milk as a function of process pressure (P) and temperature (T) (Table 2).

$$\text{Fat content} = 10.79276 - 2.279\mathrm{P} + 0.127\mathrm{T} - 0.03755\mathrm{PT} + 3.913P^2 - 8.344 \times 10^{-4}T^2 \tag{14}$$

3.2.4 | pH

It was observed that the pH values ranged from 6.4 at a pressure of 0.80 bar and a temperature of 50°C to 6.63 at a pressure of 0.60 bar and a temperature of 60°C (Table 1). The quadratic model, independent variables, interactions between them, and the LoF have not significant effect ($p > .05$) on pH. The regression coefficients of models (Table 2). Also, Equation (15) was used to calculate the pH of concentrated milk samples.

$$\mathrm{pH} = 7.268 - 0.123\mathrm{P} + 0.025\mathrm{T} - 0.020\mathrm{PT} - 0.9187P^2 + 1.327 \times 10^{-4}T^2 \tag{15}$$

For the interference between pressure and temperature as shown in the three-dimensional Figure 4d that was drawn by the response surface methodology. The value of pH was 6.53 when using pressure 0.40 bar and temperature 50°C. The value of pH decreased to 6.44 at a pressure of 0.80 bar and temperature 50°C. The value of pH also decreased at a pressure of 0.40 bar and a temperature of 60°C from 6.57 to 6.50 at 0.80 bar and a temperature of 70°C. Tanguy et al. (2019) clarified that can be expected to decrease pH during the thermal processing of milk such as concentration and sterilization processes (Tanguy et al., 2019).

3.2.5 | Ash

The results showed that the ash values ranged between 1.00% at a pressure of 0.80 bar and a temperature of 60°C to 1.43% at a pressure of 0.40 bar and a temperature of 60°C (Table 1). Probably, the pressure drop resulted in the withdrawal of the largest amount of moisture and, consequently, the ash increased with the decrease in pressure. These results are close to the findings of Madoumier el al. (2015) who found the ash content of a concentrated milk sample was 2.6%.

The quadratic model and the LoF have not significant effect ($p > .05$) on the ash (Table 1). The regression coefficients of models are shown in Table 2 and Equation (16) that used to calculate the ash of concentrated milk.

$$\begin{aligned}\mathrm{Ash} = {} & 0.89483 + 3.0273\mathrm{P} - 8.90805 \times 10^{-3}\mathrm{T} - 2.5 \times 10^{-3}\mathrm{PT} \\ & - 2.84914P^2 + 6.03448 \times 10^{-5}T^2\end{aligned} \tag{16}$$

According to Figure 4e, the ash value was decreased by increasing pressure. This may be due to an increase in temperature with an increase in the vacuum (decrease in pressure) as it led to an increase in evaporation and the withdrawal of moisture to the outside through the vacuum pump. Tanguy et al. (2019) disclosed that evaporation under vacuum depends on some factors such as thermal treatment before concentration, operation temperature, holding time in the evaporator, and storage time after concentration.

3.2.6 | Lactose

As presented in Table 1, the lactose values ranged between 8.25% at a pressure of 0.80 bar and a temperature of 60°C to 9.85% at a pressure of 0.60 bar and temperature of 60°C. These results were closed to the results of Madoumier el al. (2015) who found that the percentage of lactose in concentrated milk was 12%.

About the interference between pressure and temperature, as shown in the three-dimensional Figure 4f, the value of lactose reached 9.80% when pressure and temperature were 0.40 bar and 50°C, respectively. The lactose content was decreased to 8.41% at a pressure of 0.80 bar and temperature 50°C. Also, it decreased at a pressure of 0.40 bar and a temperature of 70°C from 9.38% to 8.94% at 0.80 bar and a temperature of 70°C. The regression coefficient illustrated in Table 2 and Equation (17) was proposed to calculate the lactose of concentrated milk.

$$\begin{aligned}\mathrm{Lact.} = {} & 16.77155 + 2.28764\mathrm{P} - 0.24941\mathrm{T} + 0.11875\mathrm{PT} \\ & - 9.73276P^2 + 1.507 \times 10^{-3}T^2\end{aligned} \tag{17}$$

3.2.7 | Total solids soluble

The results presented in Table 1 indicated that the TSS values ranged between 24% at a pressure of 0.80 bar and a temperature of 60°C and 50°C to 26% at a pressure and temperature of 0.40 bar and 50°C; 0.6 bar and 60°C; 0.4 bar and 70°C; 0.4 bar and 60°C and 0.6 bar and 50°C. This may be due to a decrease in pressure, as the vacuum can remove a large amount of moisture. Also, there was a significant effect ($p < .05$) of the linear model on the TSS. In addition, the LoF was not significant ($p > .05$). R^2 was 0.6525. These indicators showed that the linear model can be used to predict TSS values an illustrated in Equation (18):

$$\mathrm{TSS} = +32.30769 - 11.66667\mathrm{P} - 0.075000\mathrm{T} + 0.12500\mathrm{PT} \tag{18}$$

Regarding the interference between pressure and temperature as shown in the three-dimensional Figure 4i that was drawn by the response surface methodology, the value of TSS was 26.3 when using a pressure of 0.40 bar and a temperature of 50°C. The value of TSS has decreased to 24.2 when using a pressure of 0.80 bar and a temperature of 50°C. This may be due to the increase in pressure that reduced the amount of water evaporated from the milk and raised the boiling point.

3.3 | Overall color change

Table 1 shows that the ΔE values were ranged from 7.63 to 12.48 at pressure and temperature ranges of 0.40–0.80 bar and 50–70°C, respectively. At constant pressure, greater values of Δ*E* were observed at higher temperatures due to the relationship between Maillard reaction and process temperature (Kareb, Champagne, & Aïder, 2016). Also, a higher concentration of dry matter can be responsible for the color change. Furthermore, the color changes could be related to the final concentration of the product. Besides, it was observed that the effects of model were significant. Also, R-Squared was 0.9376. Therefore, the reduced cubic model (RCM) can be suggested to predict the ΔE for this RW concentration process. Δ*E* is given in Equation 19:

$$\begin{aligned}\Delta \mathrm{E} = 196.832 - 225.517\mathrm{P} - 7.306\mathrm{T} + 11.143\mathrm{PT} \\ -154.859P^2 + 0.0675T^2 + 2.23P^2T - 0.115PT^2\end{aligned} \tag{19}$$

For the interaction between pressure and temperature as shown in the three-dimensional Figure 4j which depicted by the response surface methodology, the highest value of Δ*E* was 12.63 at pressure and temperature of 0.40 bar and 70°C, respectively. Also, Δ*E* was decreased to 7.79 when process pressure and temperature were 0.80 bar pressure and 50°C, respectively. Furthermore, in general, Δ*E* was reduced by decreasing the temperature (Figure 4j). It was previously explained that reducing the milk concentration temperature can limit the changes in the overall changes of the product color due to the reduced chemical degradations and reactions such as the Maillard reaction.

3.4 | Process optimization and models validation

The results of the optimization process of the thermal performance of the RW concentrator, chemical properties, and color components are illustrated in Table 3. The result revealed that

TABLE 3 Results of the optimization process at optimum and central conditions for milk concentration using RW and CC

	RW				
	Optimum level (p = 0.40 bar; T = 69.07°C)		Central levels (p = 0.6 bar; T = 60°C)		
Dependent Variables	Experimental	Predicted	Experimental	Predicted	Experimental CC
U (W/m^2°C)	$113.21^b \pm 1.79$	113.20^b	$142.00^a \pm 3.07$	144.56^a	$98.24^c \pm 3.12$
CT (h)	$1.18^c \pm 0.07$	1.10^c	$2.00^b \pm 0.12$	2.03^b	$3.00^a \pm 0.051$
SEC (kJ/kg)	$2{,}958.39^c \pm 15.34$	$2{,}959.27^c$	$4{,}403.98^a \pm 9.65$	$4{,}406.67^a$	$3{,}952.80^b \pm 6.32$
η%	$68.14^a \pm 1.21$	68.15^a	$46.65^b \pm 1.98$	48.15^b	$30.25^c \pm 3.86$
MC%	$74.61^b \pm 0.30$	74.23^b	$74.00^c \pm 3.56$	74.69^c	$75.00^a \pm 0.01$
Pr%	$7.43^b \pm 0.04$	7.59^b	$7.13^c \pm 1.02$	7.26^c	$7.70^a \pm 0.26$
Fat%	$7.45^a \pm 0.07$	7.43^a	$7.66^a \pm 0.09$	7.47^a	$7.20^b \pm 0.05$
Ash%	$1.25^b \pm 0.09$	1.30^b	$1.42^a \pm 0.01$	1.28^a	$1.11^c \pm 0.06$
Lact.%	$9.24^b \pm 0.17$	9.50^b	$9.79^a \pm 0.96$	9.38^a	$9.00^c \pm 0.15$
TSS%	$25.97^a \pm 0.14$	26.00^a	$26.00^a \pm 1.08$	25.97^a	$25.00^b \pm 0.42$
pH	$6.51^a \pm 0.078$	6.48^a	$6.60^a \pm 0.12$	6.54^a	$6.47^b \pm 0.03$
Δ*E*	$9.49^c \pm 0.64$	9.58^c	$11.44^a \pm 0.15$	11.26^a	$10.77^b \pm 1.23$

The different l letters in the rows indicate a significant effect at level of 0.05.

Abbreviations: CC, conventional concentration; CT, concentration-time; MC, moisture content; Lact., lactose; P, pressure; Pr., protein; RW, refractance window; SEC, specific energy consumption; SS, sum of square; T, temperature; TSS, total soluble solids; U, heat transfer coeffecient; η, energy effeciency; Δ*E*, total color change.

the optimum milk concentration process conditions were a temperature of 69.07°C and a pressure of 0.43 bar. These conditions provide a concentrated milk with predicted U (113.20 W/m^2°C), CT (1.1 hr), SEC (2,959.27 kJ/kg), η (68.15%), moisture content (74.23%), protein (7.59%), fat (7.43) ash (1.30%), lactose (9.50%), TSS (26.00%), pH (6.48) and ΔE (9.58).

To check the veracity of the predicted values by models at optimized conditions, validation experiments were performed based on the central and optimum concentration process with 6 replications. As illustrated in Table 3, when milk concentrated by optimum or central conditions, the experimental results were similar to the predicted results by using optimized models, which verified that the validity of response regression models. This reflects the actual results of the independent variables. Moreover, there is a significant difference ($p < .05$) between the experimental data for the optimum level and the experimental data for the central level. The U, η, fat, ash, lactose content, TSS, and pH for milk concentrated by CC were lower than those of optimized and central levels of RW. Furthermore, the CT, and MC of milk concentrated by CC were higher than both optimized and central levels of RW. This study demonstrated that the RW concentrator system possesses higher efficiency and thermal performance could significantly improve the concentrated milk quality.

4 | CONCLUSIONS

RW was found to be superior to the conventional concentration method of milk concentration in terms of processing time, energy consumption, energy efficiency, and some quality parameters of the product. This innovative technique saved about 61% of the processing time and reduced the changes in the product color during concentration. Also, it was revealed that selecting the appropriate pressure and temperature can enhance the performance of the RW system and product characteristics. The food industry may benefit from such emerging approaches in the future after further evaluations including up-scaling studies.

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CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

NOMENCLATURE

a^*	Redness-greenness
Adeq	Adequate precision
Adj	Adjusted
ANOVA	Analysis of variance
b^*	Yellowness-blueness
CVS	Computer vision system
CC	Conventional concentration
CIE	Commission international d'eclairage
CP	Specific heat (kJ/kg.°C)
Cpi	Specific heats of milk composition (kJ/kg.°C)
CT	Concentration-time
D	Desirability
E	Overall color
F	Fat (%)
L*	Lightness
M	Mass flow rate of milk (kg/s)
LoF	Lack of Fit
LMTD	Logarithmic mean temperature difference
r	Impotence
R	Correlation coefficient
RCM	Reduced cubic model
RM	Raw milk
RSM	Response surface methodology
RW	Refractance window
SEC	Specific energy consumption
SS	Sum of squares
Std. Dev.	Standard deviation
T	Temperature (°C)
TSS	Total soluble solid (%)
U	Overall heat transfer (W/m^2°C)
x	Value

Greek symbols

β	Constant
γ	Constant
Δ	Differences
η	Energy efficiency
λ_w	Latent heat (kJ/kg)

Subscripts

i	Linear
i	Response
ii	Quadratic
h0	Hot water
m0	Hot milk
m	Milk
m	Mean temperature
mi	Cold milk
hi	Cold water
o	Interceptpre.Predicted

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