



A Survey of Effective Parameters in Biomass Separation Using Vacuum Membrane Filtering: A Case Study of Pectin Acidic Solution [†]

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Abstract: Pectin, which is made from citrus peel and waste, is one of the most commonly used compounds in the food industry. For large-scale production, a combination of membrane-vacuum filtering has been suggested as an alternative to traditional methods of purifying the acidic solution for pectin extraction. This study investigates the main factors involved in the membrane filtering system for the separation of fibrous materials from an acidic pectin solution under vacuum. These factors include filter aid particle size, the amount of filter aid (perlite) added to the solution, and the vacuum level. They affect separation quality, volumetric flow rate, and energy consumption. A vacuum separation device was developed for this purpose to separate the fibrous material dissolved in the solution. The independent variables were examined at three levels, and the data were analyzed. The optimum value for each variable was determined using the response surface method (RSM). Results revealed that increasing the vacuum level from 0.2 to 0.4 bar increases the flow rate 6.5-fold, while further increase in the vacuum level decreases the flow rate. This indicates clogging of the paper filter and decreased flow rate at a vacuum level of 0.6 bar and perlite particle size of 100 microns. The evaluation results showed that the thickness of the perlite layer has the greatest effect on the separation efficiency. When increased from 1 to 2 cm, it increases the efficiency 2.5-fold. The maximum value of separation efficiency was obtained at a vacuum level of 0.2 bar, a particle size of 20 microns, and a perlite thickness of 2 cm. The energy consumption of 60-micron perlite was 0.74 Wh in the optimal state, while the larger and smaller sizes of perlite had 4.5 times the energy consumption. These findings are applicable in the industrial-scale implementation of a biomaterial separation system using vacuum membrane filtering.

Keywords: extraction; yield; energy consumption; orange peel; perlite



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1. Introduction

Separation is a critical process in engineering, especially when dealing with fluids containing vastly different components. This process is also crucial in the food industry and related equipment [1]. Separation techniques include liquid–solid, solid–gas, and solid–solid, which are carried out using several methods, including centrifugation, sedimentation, disk filtration, positive pressure, and vacuum filtration [2]. In one of the techniques used for solid–liquid separation, a suspension passes through a porous medium that retains solid particles. To achieve this, a driving force—such as positive pressure, negative pressure (vacuum), gravity, or centrifugal force—is applied to force the fluid through the medium [3].

Vacuum filtration is a process in which a liquid passes through a filter medium, such as a paper or cloth filter, where solid particles are trapped and form a cake that is removed

by applying vacuum pressure [4]. In vacuum filters, as the liquid approaches the filter medium, solid particles settle on the surface of the filter, forming a cake, while the liquid passes through the medium due to the negative pressure created by the vacuum. The initial layer of cake, formed by perlite, acts as a filter medium, and more solid particles are deposited on it, increasing its thickness as the liquid continues to pass through. Large industrial processes often rely on continuous filters [5]. Rotary vacuum drum filters, such as the one shown in Figure 1, are the most commonly used filtration devices in the industry.

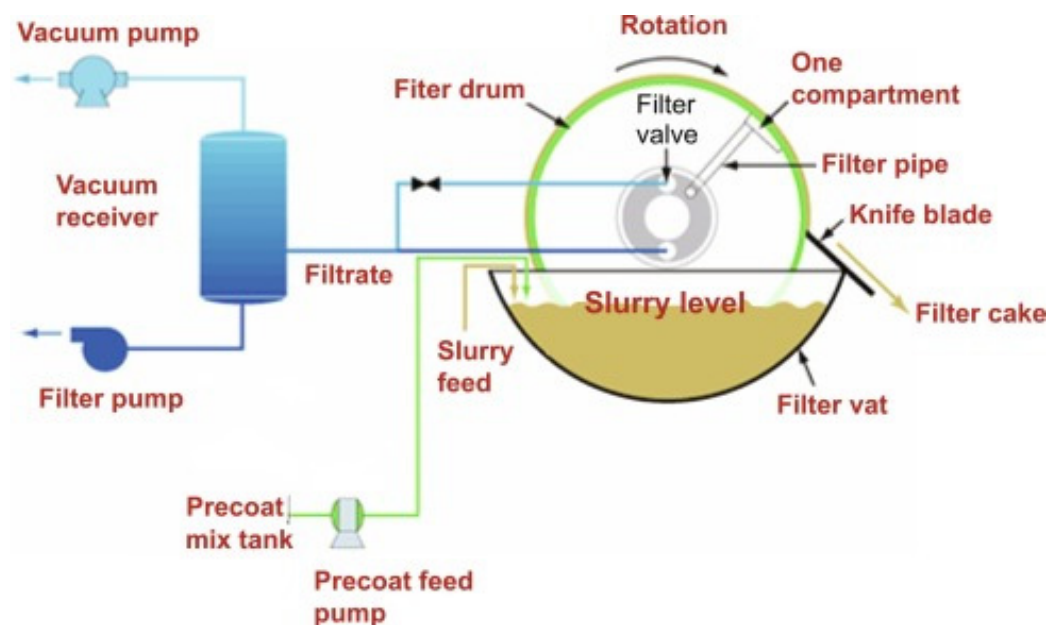


Figure 1. Rotary vacuum drum filter schematic [5].

A rotary vacuum drum filter (RVDF), first introduced in 1872, is one of the oldest filters used in the separation of solid and liquid materials in the industry. The rotary vacuum drum filter consists of a filter drum covered by a filter or cloth. Other components include a vacuum pump, a fluid storage tank, and a filter aid [6]. The rotary vacuum drum filter is used in the pharmaceutical industry to collect calcium carbonate, magnesium carbonate, and starch and separate mycelium for the production of antibiotics. On the other hand, it is used in the food industry to filter fluids with a significant amount of solid material that requires continuous filtration [7]. Common problems with vacuum filters include premature clogging and settling of solid materials in the fluid storage tank (Figure 2). Many engineering variables play a vital role in vacuum filters, but they may vary depending on the filtered fluid type. Important variables include the cake layer thickness, the filter aid particle size, and the vacuum pressure required for filtration [8].

Pectin is an acidic hydrocolloid compound with many applications as a natural additive in various industries, especially in food, pharmaceuticals, and medicine. In the food industry, it is used for its gelling, stabilizing, texture-creating, emulsifying, thickening, and fat-replacing features. Additionally, in medicine and pharmaceuticals, it serves as a dietary fiber for digestive treatment. The most common use of pectin in the food industry is in the production of jam and jellies as a gelling and thickening agent [9,10]. Pectin is present in all plants, but its amount and chemical properties vary depending on the plant species, variety, maturity, plant part, tissue, and growth conditions [11]. Regarding its medical applications, pectin is particularly useful in preventing colon cancer and reducing blood cholesterol levels [12]. There are various methods for pectin extraction.

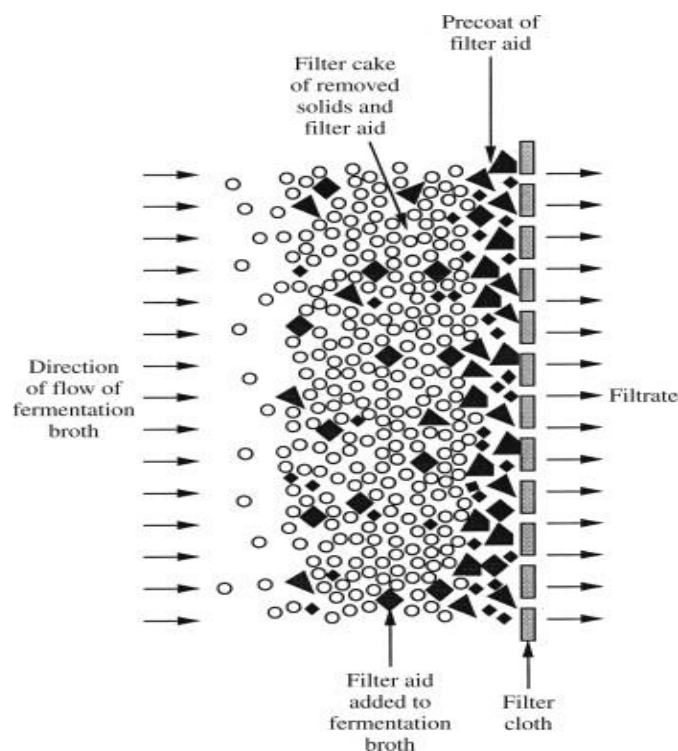


Figure 2. How filter assistance operates [10].

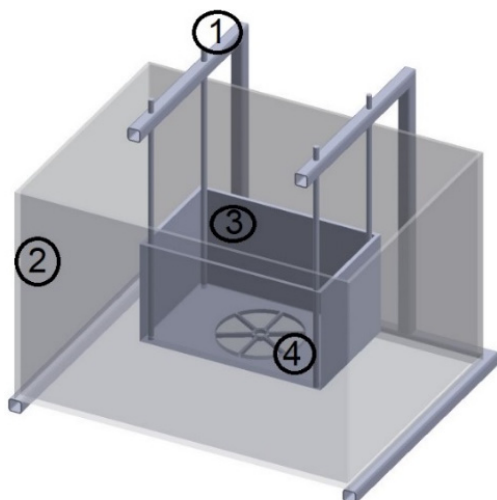
The industrial method for pectin extraction involves using acidic solutions at high temperatures. Traditionally, pectin is extracted by continuously stirring it in an acid solution at a temperature of 80 to 100 degrees Celsius for one hour. Pectin extraction depends on various factors, including temperature, pH level, solvent properties, solid-to-solvent ratio, dried solid materials, particle size, and diffusion rate [13].

Given the mentioned applications of pectin, the increasing importance of productivity improvement in the food industry—especially in the field of recycling crucial materials such as pectin extraction—and the lack of scientific reports on the use of vacuum filters in separating such fluids have been the motivation behind this research. On an industrial scale, solid–liquid separation is performed using a combination of vacuum and membrane filters. However, regarding the important variables in vacuum separation—variables that significantly impact the quality and efficiency of separation, and upon which fluid impurities heavily depend—no report has been observed. Therefore, in this study, the important variables of vacuum filters in separating fibrous materials from acidic pectin solutions were investigated. The dependent variables included the size of the filter aid particles, the thickness of the filter aid layer, and the vacuum level of the filter. All of these factors affected the dependent research variables, namely energy consumption, production yield, flow rate, and separation quality.

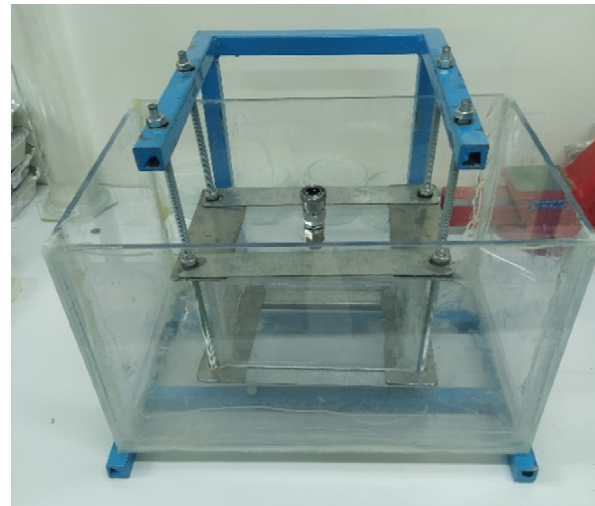
2. Methods and Materials

2.1. Design and Construction Method of a Vacuum Filter System

In order to simplify the components and apply dependent variable surfaces, a small-scale experimental vacuum filter system was constructed as described below. The trend chart of the rotary vacuum filter system is visible in Figure 3, which is explained further below. In order to provide vacuum levels, a vacuum pump was used, which could be adjusted using a gate valve at those vacuum levels.



(a)



(b)

Figure 3. Vacuum-operated filter system. (a): Software design was made, (b): a test system was constructed. 1: holding chassis, 2: fluid storage tank, 3: vacuum tank, 4: storage valve for design templates, and holding chassis.

In order to utilize different sizes of filter aids (perlite) for separation, perlite with a specific size was used at three levels of less than 20 microns, 40 to 60 microns, and 60 to 80 microns. Also, the variable thickness of the layer was considered at three levels of 1, 1.5, and 2 cm. Templates were made and used according to Figure 4 for precise application of these sizes.

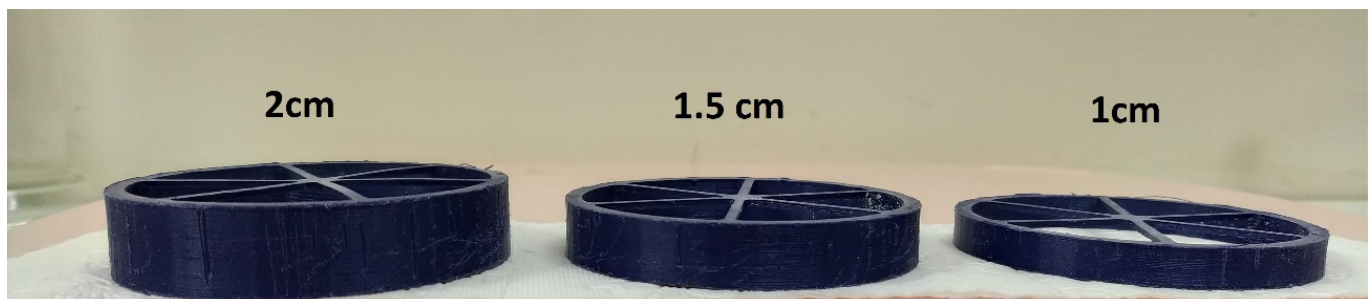


Figure 4. Three different stencil thicknesses: 2, 1.5, and 1 cm.

In order to maintain the prepared fluid and the vacuum chamber, two tanks were designed in Solidworks 2016 software and then made of Plexiglas using sheet metal. Finally, to prevent possible leaks, aquarium glue was used. Considering that solid materials in the fluid are insoluble, it is obvious that they will eventually settle. Therefore, a mechanical stirrer was used.

In order to connect the vacuum pump and the vacuum chamber, pneumatic connections were used. The system of the vacuum separator is shown in Figure 5, considering the various components and their connections. In this system, different parts of the supporting chassis are used to hold the system and the vacuum pump to create a vacuum force for filtration, as well as two tanks for storage and vacuum reservoir.

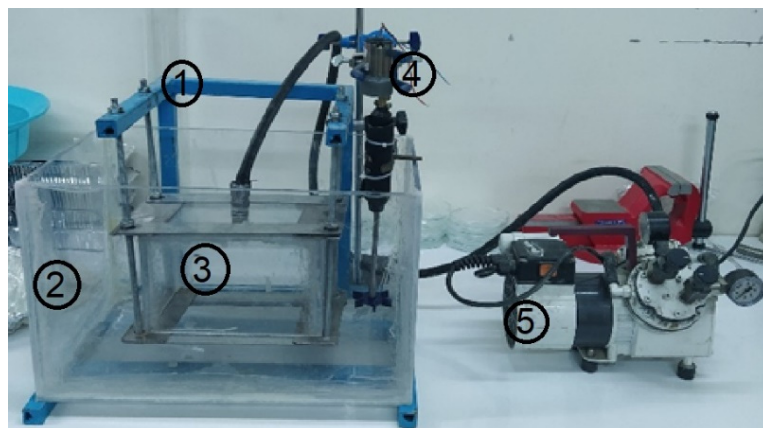


Figure 5. Vacuum-built filter system. 1: holding chassis, 2: fluid storage tank, 3: vacuum tank, 4: mechanical stirrer, and 5: vacuum pump.

2.2. Method of Preparing Filtration Fluid

To create orange peel powder, 40 kg of fresh Thomson oranges were procured from the fruit and vegetable market in Tehran. The peels were carefully separated for the drying process. Subsequently, the orange peels were placed in an electric oven and dried at a temperature of 45 degrees Celsius for 24 h. After drying, the peels were ground using a mill and then sieved through a mesh number 40 sieve to ensure consistent particle size. The resulting powder was stored in a refrigerator at a temperature of 4 degrees Celsius in preparation for extraction. To prepare the solution, the orange powder was mixed with distilled water in a ratio of 1 to 25. The pH of the solution was adjusted to 7.1 using hydrochloric acid. The mixture was thoroughly blended using a mixer for 50 min at a temperature ranging between 80 and 82 degrees Celsius (as shown in Figure 6). Once the solution was ready, it underwent filtration through a cloth filter and was subsequently cooled to room temperature. The resulting liquid served as the substance under investigation in this study. In contrast to the standard laboratory process, which involves centrifugation to separate pulp from liquid, our study employed centrifugation and vacuum filtration for the separation stage. Additionally, pectin was extracted from the liquid using ethanol in a ratio of 1 to 3.



Figure 6. A view of the sample solution preparation process using a stirrer.

2.3. Method of Filtration Using a Vacuum Filtration System

The vacuum filtration process involved several steps. Initially, a specific volume of distilled water and perlite was introduced into the tank to create the filter aid layer. Once the desired thickness of the filter aid was achieved, the pump was turned off, and adjustments were made to the perlite layer thickness. To ensure precise control, a three-dimensional structure created using coating technology was employed. Next, a predetermined amount of the prepared fluid was poured into the tank, along with the specified perlite layer thickness. By setting the desired vacuum level, the vacuum pump was activated, allowing the solution to pass through both the filter aid and paper filter. The separation time was carefully recorded during this stage. In the final step, the filtered solution was diluted with 96% ethanol at a ratio of 1:3. The mixture was then stored at 4 °C for 12 h. Using a centrifuge operating at 10,000 revolutions per minute for 20 min, the pectin—present as cloudy particles in the water-alcohol mixture—was separated. Finally, the obtained pectin underwent drying in an electric oven for 6 h. This study evaluated the impact of varying vacuum levels (6.0, 4.0, 2.0 bar), perlite layer thickness (2, 1.5, 1 cm), and perlite particle size (100, 60, 20 microns) on energy consumption, flow rate, separation efficiency, and pectin extraction efficiency.

2.4. Method of Measuring Energy Consumption

The method for measuring energy consumption: In order to measure the energy consumption of each experiment, a power analyzer was used to record the power consumption at the selected vacuum levels of 6.0, 4.0, and 2.0 bar. Based on the recorded time for each experiment, the energy consumption was calculated.

2.5. Method of Measuring Production Flow Rate

Considering that the volume of the desired solution was measurable and known, and on the other hand, the separation process time was recorded, the average volumetric flow rate was easily calculated using Equation (1).

$$Q = \frac{v}{t} \quad (1)$$

In this regard:

Q = Volumetric flow rate (mL/s)

v = Volume of fluid (mL)

t = Time (s)

2.6. Method of Measuring Separation Efficiency

Separation efficiency measurement method: A specific volume of the sample solution was taken and poured into an aluminum foil and shaped. Then, it was dried for 24 h at a temperature of 105 degrees Celsius in an electric oven. Similarly, this process was repeated for the filtered solution and the dry matter was measured using a balance in the end. By using Equation (2), the separation efficiency was calculated.

$$Ra_f = \frac{m_t - m_i}{m_t} * 100 \quad (2)$$

In this regard:

Ra_f = Separation efficiency (%)

m_t = Amount of solid material in the control solution (g)

m_i = Amount of solid material in the filtered solution (g)

2.7. Statistical Analysis

Statistical analysis: The number of experiments performed was 17, with 5 repetitions for the center point. Table 1 shows the treatments applied in the experiment for analysis using response surface methodology and their corresponding responses.

Table 1. Treatment strategies used in the experiment for the response surface analysis.

Number	Pressure (bar)	Perlite Particle Size (micron)	Perlite Layer Thickness (cm)
1	0.4	60	1.5
2	0.4	60	1.5
3	0.4	60	1.5
4	0.4	60	1.5
5	0.2	60	2
6	0.2	20	1.5
7	0.2	60	1
8	0.4	100	2
9	0.6	60	2
10	0.4	20	2
11	0.4	20	1
12	0.6	60	1
13	0.6	20	1.5
14	0.4	100	1
15	0.6	100	1.5
16	0.2	100	1.5
17	0.4	60	1.5

In this study, to analyze the effect of independent variables on dependent variables, Expert Design software (<https://www.statease.com/software/design-expert/>, accessed on 1 November 2023) and the Box–Behnken method were used in the general method of response surface analysis.

3. Results and Analysis

3.1. Results of Variance Analysis of Energy Consumption Data by Vacuum Filtration System

The ANOVA results for energy consumption data obtained from the vacuum filtration system are summarized in Table 2. The study findings reveal that the model is statistically significant, and there is no significant lack of fit. Consequently, the chosen model and analyses are both reliable and valid. At the 1% significance level, the effects of vacuum level and particle size squared are significant. Additionally, at the 5% probability level, the effects of layer thickness of perlite, vacuum level squared, and layer thickness of perlite are also significant. These significant effects underscore the importance and influence of the selected independent variables in this experiment. Specifically, the effects of vacuum level (A), particle size of perlite (B), and layer thickness of perlite (C) are detailed in Table 2.

Using the response surface methodology, a complete second-order polynomial model with a determination coefficient of 0.97 was selected to estimate the energy consumption by varying the levels of independent variables. The proposed actual model is a second-degree polynomial function represented by Equation (3).

$$\text{Log}_{10}^E = 1.92 - 8.15 \times A - 0.03 \times B + 1.61 \times C - 0.008 \times A \times B + 0.1 \times A \times C - 0.003 \times B \times C + 8.54 \times A^2 + 0.0003 \times B^2 - 0.55 \times C^2 \quad (3)$$

The variable E represents energy consumption in this equation, and based on the results of the regression model, the proposed model for predicting energy consumption with the selected independent variables is significant. The positive sign of each term indicates a synergistic effect, and the negative sign indicates a negative effect of the variable(s) on the response.

Table 2. Variance analysis of the tested variables for energy consumption.

Source	df	Sum of Squares	Mean Squares	F-Value	p-Value
Model	9	3.01	0.3343	26.93 ^a	0.0001
A—Pressure	1	0.8902	0.8902	71.72 ^b	0.0001<
B—Perlite particle size	1	0.0701	0.0701	^a 5.65	0.0491
C—Perlite layer thickness	1	0.0714	0.0714	1.46 ^a	0.0476
B × A	1	0.0181	0.0181	^{ns} 1.08	0.2668
C × A	1	0.0004	0.0004	^{ns} 0.0327	0.8615
C × B	1	0.0169	0.0169	^{ns} 1.46	0.2820
A × A	1	0.4919	0.4919	^a 39.63	0.0004
B × B	1	1.33	1.33	106.96 ^b	0.0001<
C × C	1	0.0789	0.0789	^a 6.35	0.0398
Residual	7	0.0869	0.0124		
Lack of fit	3	0.0706	0.0235	^{ns} 5.77	0.0617
Pure error	4	0.0163	0.0041		
Cor total	16	3.1			

^a significant at the 5% probability level. ^b significant at the 1% probability level. ^{ns} not significant.

3.2. The Interaction Effect of Independent Variables on Energy Consumption

In Figure 7, contour lines representing surface response charts for the dependent variable (energy consumption) illustrate variations in the independent variables. Analyzing Figure 7a, we observe that the minimum energy consumption occurs at a vacuum level of 0.4 bar. At this minimum vacuum level, energy consumption changes proportionally with the increase in perlite particle size, reaching its lowest value for particles measuring 60 microns. When considering both maximum and minimum perlite particle sizes, an increase in vacuum level leads to a significant reduction in energy consumption, although the trend becomes less pronounced. This behavior is attributed to shorter fluid passage times (assuming constant volume) at higher vacuum levels, resulting in decreased energy consumption. Conversely, at the intermediate level of perlite particle size and the minimum vacuum level, the filtration process performs well. In this case, the particle size is appropriate, preventing filter clogging. Consequently, energy consumption in this scenario is lower than in other cases. Furthermore, gradually increasing the thickness of the perlite layer at the minimum vacuum level (as shown in Figure 7b) initially raises energy consumption slightly, followed by a subsequent decrease.

The decrease in energy consumption observed in this scenario highlights the impact of thinner perlite layers. When the auxiliary filter absorbs less solid fluid material, filter clogging occurs more rapidly, leading to increased filtration time and subsequently higher energy consumption. However, by increasing the thickness of the auxiliary filter, solid material absorption improves, resulting in a more efficient filtration process and a downward trend in energy consumption. Conversely, elevating the vacuum level reduces filtration time, ultimately leading to decreased energy consumption. In other conditions, the trend of energy consumption change is insignificant. As depicted in Figure 7c, overall changes in energy consumption lack statistical significance. Nevertheless, it is worth noting that the trend of energy consumption is partly proportional to variations in perlite particle size. When using 20-micron perlite, solid particles struggle to pass through the auxiliary filter, prolonging the filtration process. Conversely, with 100-micron perlite, solid fluid particles pass through the filter more easily, causing faster clogging and increased energy consumption. A study by Manu Huttunen et al. [14], investigating energy consumption in a vacuum filter system by examining vacuum levels of 6.0, 4.0, and 2.0 bar, revealed that changes in vacuum levels do not significantly impact energy consumption. Optimal energy consumption occurs at vacuum levels of 2.0 to 3.0 bar.

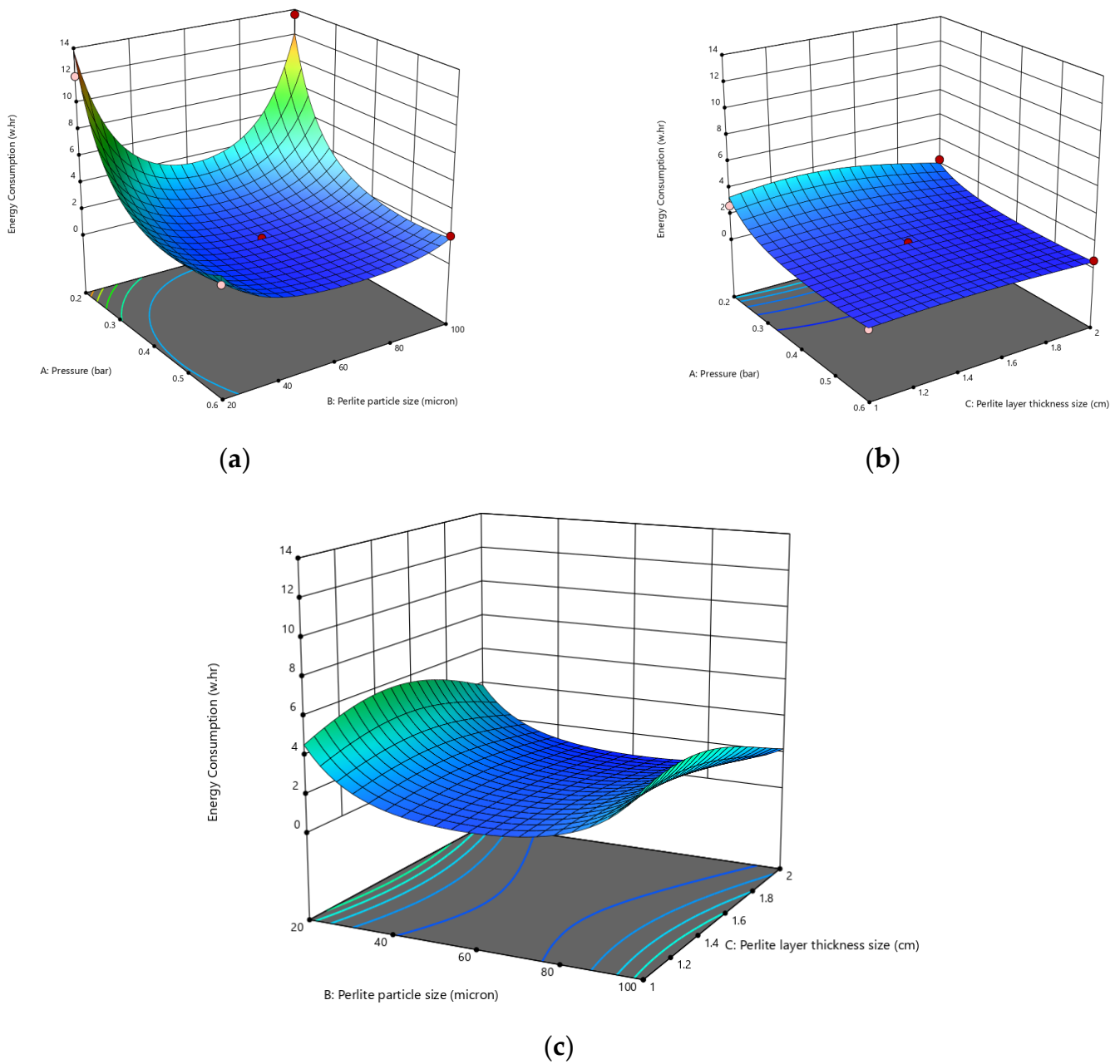


Figure 7. The effect of different variables on energy consumption (a). Reaction of pressure and perlite particle size (b). Reaction of pressure and perlite layer thickness (c). Reaction of perlite particle size and perlite layer thickness.

3.3. ANOVA Results for Fluid Flow Rate

The fluid permeability represents the specific volume of fluid passing through the filter section per unit of time. The ANOVA results, as shown in Table 3, indicate that the model is statistically significant, and there is no significant lack of fit. Consequently, the chosen model and analyses are both reliable and valid. At a 5% probability level, the effects of vacuum level, vacuum level squared, perlite particle size, and perlite layer thickness are significant. These significant effects underscore the importance and impact of the selected independent variables in this experiment. Specifically, the effects of vacuum level (A), perlite particle size (B), and perlite layer thickness (C) are detailed in Table 3.

Table 3. Variance analysis of the tested variables for flow rate.

Source	df	Sum of Squares	Mean Squares	F-Value	p-Value
Model	9	3.07	0.3414	17.96 ^a	0.0005
A—Pressure	1	0.9039	0.9039	47.55 ^a	0.0002
B—Perlite particle size	1	0.0196	0.0196	^{ns} 1.03	0.3442
C—Perlite layer thickness	1	0.0173	0.0173	^{ns} 0.9102	0.3718
B × A	1	0.021	0.021	^{ns} 1.1	0.3285
C × A	1	0.0001	0.0001	^{ns} 0.0044	0.9488
C × B	1	0.0065	0.0065	^{ns} 0.3415	0.5773
A × A	1	0.7255	0.7255	^a 37.17	0.0005
B × B	1	1.22	1.22	^b 64.03	0.0001<
C × C	1	0.1203	0.1203	^a 6.33	0.0401
Residual	7	0.1331	0.019		
Lack of fit	3	0.1103	0.0368	6.45 ^{ns}	0.0518
Pure error	4	0.0228	0.0057		
Cor total	16	3.21			

^a significant at the 5% probability level. ^b significant at the 1% probability level. ^{ns} not significant.

The full second-degree polynomial model with an R-squared value of 85/95 was selected using the response surface methodology to estimate the value of the flow rate by changing the values of the independent variables. The proposed actual model is a second-degree polynomial function represented by Equation (4).

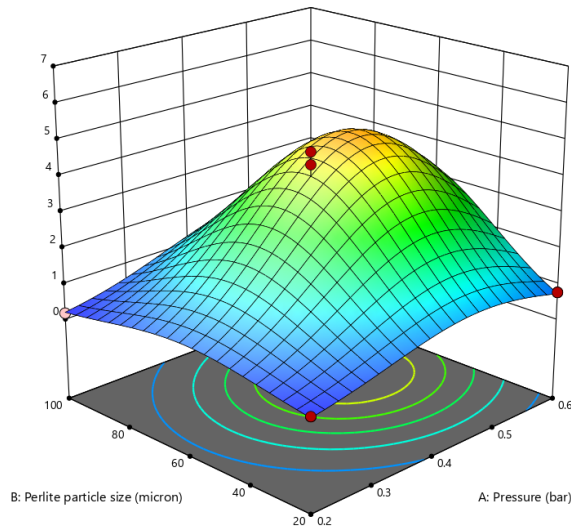
$$\begin{aligned} \text{Log}_{10}^q = & -1.28 + 9.51 \times A + 0.03 \times B - 2.04 \times C + 0.009 \times A \times B \\ & - 0.46 \times A \times C + 0.002 \times B \times C - 10.38 \times A^2 + 0.0003 \times B^2 \\ & + 0.68 \times C^2 \end{aligned} \quad (4)$$

Based on the results of the regression model, the proposed model for predicting the trend of fluid flow with the selected independent variables is significant. The positive sign of each term indicates a positive effect on the response, and the negative sign indicates a negative effect of the variable(s) on the response.

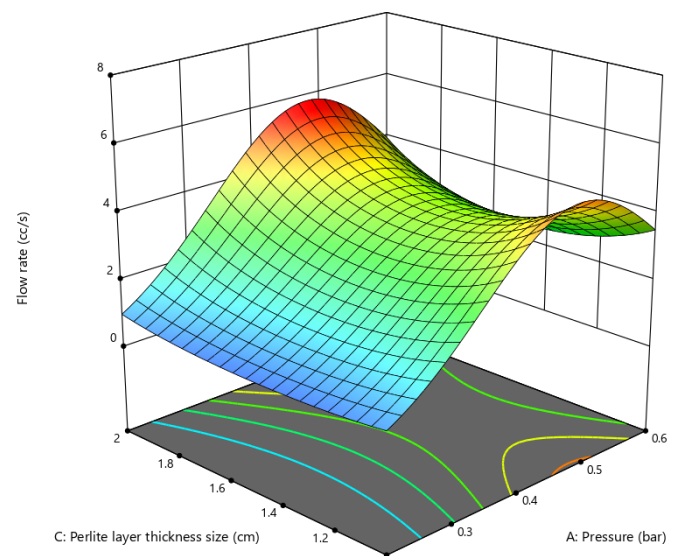
3.4. The Mutual Effect of Independent Changes on Discharge

In Figure 8, the response surface diagram of the dependent variable (flow rate) against changes in the vacuum level and size of perlite particles can be seen in both three-dimensional and contour line forms. According to Figure 8a, which shows the response surface diagram of the dependent variable (flow rate) against changes in the vacuum level and size of perlite particles in the form of contour lines, an increase in the size of perlite particles corresponds to proportional changes in the flow rate. This trend becomes more pronounced with an increase in the vacuum level. Similarly, when the size of perlite particles is held constant, the changes in the dependent variable (flow rate) are proportional to an increase in the vacuum level. The maximum volumetric flow rate (fluid passing through the filter section) was achieved at a vacuum level of 5.0 bar and a particle size of 60 microns. In cases of maximum and minimum particle size, due to the rapid clogging of the filter section or the inability of the fluid to pass through the auxiliary filter due to the small size of the particles, the dependent variable is lower at the lower level. Considering the reasons mentioned, the intermediate size of perlite particles is suitable. On the other hand, the trend of change in the vacuum level on the dependent variable shows that at maximum vacuum level, with a faster flow of fluid, the possibility of clogging increased both in the auxiliary filter and in the filter section. Therefore, a decrease in the flow rate passing through the filter section was observed at a vacuum level of 6.0 bar. Based on Figure 8b, at a vacuum level of 2.0 bar and with an increase in the thickness of the perlite layer, the trend of change in the dependent variable is slight. However, with an increase in the vacuum level, a partially increasing trend was observed. On the other hand, con-

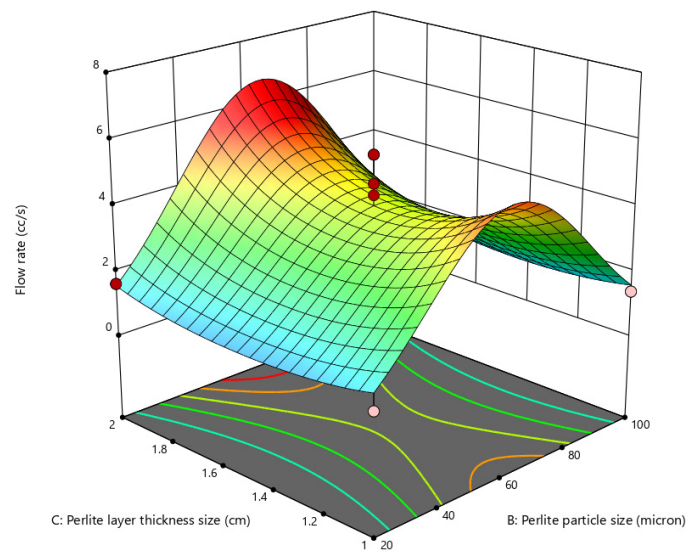
sidering a specific thickness of the support filter, with an increase in the vacuum level, the increasing–decreasing trend of the dependent variable was observed. Since the maximum amount of fluid flow passing through the filter cross-section is desired, this condition was achieved at the maximum thickness of the support filter (cm^2) and the intermediate vacuum level (4.0 bar). As expected, with an increase in the thickness of the support filter, the possibility of solid particles adhering to the support filter decreased, so the maximum particle size of the perlite layer is desired.



(a)



(b)



(c)

Figure 8. The effect of different variables on the flow rate (a). Reaction of pressure and perlite particle size (b). Reaction of pressure and perlite layer thickness (c). Reaction of perlite particle size and perlite layer thickness.

As shown in Figure 8c, with an increase in the independent variables, the dependent variable (flow rate) varies partially. As mentioned in previous sections, with smaller perlite particle sizes, the density is higher, and the fluid passes through the perlite with

difficulty. On the other hand, in the maximum particle size of perlite, with the increase in the defects inside the support filter, the possibility of adhering to the filter cross-sectional area increased, and the filtration process was not well-performed, resulting in a decrease in the dependent variable. In cases of maximum and minimum particle sizes of perlite, the trend of change in the dependent variable is negligible with an increase in the thickness of the perlite layer. However, in intermediate particle sizes of perlite, with a specific size considered, the dependent variable follows a partially increasing trend with an increase in the thickness of the perlite layer. Considering that the maximum amount of fluid flow passing through the filter cross-section is desired, the maximum thickness of the perlite layer and the intermediate particle size of perlite are the optimal points for maximizing the dependent variable. Other researchers report that with an increase in pressure, the filtration rate increases, but the relationship between pressure and the amount of fluid passing through the line is of a linear type.

3.5. Results of Analysis of Variance (ANOVA) for Separation Efficiency

The separation efficiency is defined as the ratio of the difference between the amount of solid material in the witness state and the amount of solid material separated by the vacuum filter system to the amount of solid material separated in the witness state. The results of the analysis of variance (ANOVA) for the effect of different variables on separation efficiency are presented in Table 4. The findings of this study indicated that the model is significant and the lack of fit is not significant. Therefore, the selection of models and analyses is reliable and valid. The effects of particle size, layer thickness, and the square of particle size of perlite are significant at a 1% level of significance. The effects of vacuum level, the interaction of vacuum level and particle size of perlite, vacuum level and layer thickness of perlite, as well as the square of vacuum level are significant at a 5% level of significance. The significance of the mentioned effects indicates the importance and impact of the selected independent variables in this experiment. The effects of vacuum level, particle size of perlite (labeled as A), and layer thickness of perlite (labeled as C) are shown in Table 5.

Table 4. Variance analysis of the tested variables for extraction yield.

Source	df	Sum of Squares	Mean Squares	F-Value	p-Value
Model	9	3332.33	370.26	132.24 ^b	0.0001<
A—Pressure	1	168.54	168.54	60.19 ^a	0.0001
B—Perlite particle size	1	940.48	940.48	335.89 ^b	0.0001<
C—Perlite layer thickness	1	1609.71	1609.71	^b 574.9	0.0001<
B × A	1	41.54	41.54	^a 14.84	0.0063
C × A	1	82.54	82.54	^a 29.48	0.0010
C × B	1	0.8742	0.8742	^{ns} 0.3122	0.5937
A × A	1	16.08	16.08	^a 6.00	0.0442
B × B	1	460.44	460.44	^b 164.44	0.0001<
C × C	1	5.36	5.36	^{ns} 1.91	0.2092
Residual	7	19.6	2.8		
Lack of fit	3	8.17	2.72	0.9531 ^{ns}	49.54
Pure error	4	11.43	2.86		
Cor total	16	3351.93			

^a significant at the 5% probability level. ^b significant at the 1% probability level. ^{ns} not significant.

Table 5. Independent and objective program boundary conditions to optimize the filtration process.

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight
Pressure	is in range	0.2	0.6	1	1
Perlite particle size	is in range	20	100	1	1
Perlite layer thickness	is in range	1	2	1	1
Energy consumption	minimize	0.54	13.6	1	1
Flow rate	maximize	0.17	5.62	1	1
Extraction yield	maximize	18.46	68	1	1

Using the response surface method, a full second-degree polynomial model with a determination coefficient of 42/99 was selected to estimate the value of separation efficiency by varying the levels of independent variables in the coded form. The proposed actual model to examine the effects of selected variables is a second-degree polynomial function, as represented by Equation (5) ($42/99 = R^2$).

$$Ra_s = 23.14 - 18.93 \times A - 1.25 \times B + 58.67 \times C + 0.4 \times A \times B - 45.42 \times A \times C + 0.02 \times B \times C + 49.93 \times A^2 + 0.006 \times B^2 - 4.5 \times C^2 \quad (5)$$

The positive sign of each term indicates a positive effect, and the negative sign indicates a negative effect of the variable(s) on the response. It can be seen that the maximum impact is due to the thickness of the perlite layer and the minimum effect is due to the square of the vacuum level. Based on the results of the regression model, the proposed model is meaningful in predicting the trend of flow rate with the selected independent variables.

3.6. The Interaction Effect of Independent Variables on Separation Efficiency

In Figure 9, the response surface graphs of the dependent variable (extraction yield) against changes in the level of vacuum and the size of perlite particles are shown as contour lines. In Figure 9a, the response surface graph of the dependent variable (separation yield) against changes in the level of vacuum and the size of perlite particles is shown. Based on these figures, it can be observed that the trend of the dependent variable, separation yield, is not significant at maximum perlite particle size with an increase in vacuum level, but gradually increases with a decrease in perlite particle size for a specific vacuum level. It is notable that with smaller perlite particle sizes, more solid materials are absorbed by the filter aid, thus increasing the separation yield. On the other hand, with an increase in the vacuum level, more solid materials pass through the filter aid, resulting in a lower separation yield, and vice versa. At minimum vacuum level, the maximum separation yield is achieved. Therefore, the maximum separation yield is achieved at a vacuum level of 2.0 bar and a perlite particle size of 20 microns. According to Figure 9b, the separation efficiency gradually increases with an increase in the thickness of the perlite layer for a specific vacuum level, and this trend has a steeper slope at lower vacuum levels. With an increase in the suction force at higher vacuum levels, more solid materials pass through the filter aid, resulting in a decrease in the trend of variable-dependent separation efficiency. On the other hand, it should be noted that the change in the thickness of the perlite layer is not significant in the minimum state.

As Figure 9c shows, with an increase in the thickness of the filter aid layer for a specific size of perlite particles, the trend of variable-dependent separation efficiency is increasing. On the other hand, the trend of separation efficiency change with a specified thickness of the filter aid layer is also bilinear. Although the maximum separation efficiency is desirable, it is achieved at the minimum size of perlite particles (20 microns) and thickness of the perlite layer (2 cm). On the other hand, the parallel lines demonstrate the bilinear relationship between the size of perlite particles and the variable-dependent separation efficiency. Additionally, Davarzani et al. [15] showed, in a study comparing the effect

of filter size on separation performance, that smaller filter particle size leads to better separation in the purification process. Furthermore, Filadieu et al. [16] demonstrated that reducing the size of the pores in the membrane filtration process leads to the formation of a layer of carbohydrates and proteins on the filter pores, indicating greater separation efficiency for smaller particle sizes.

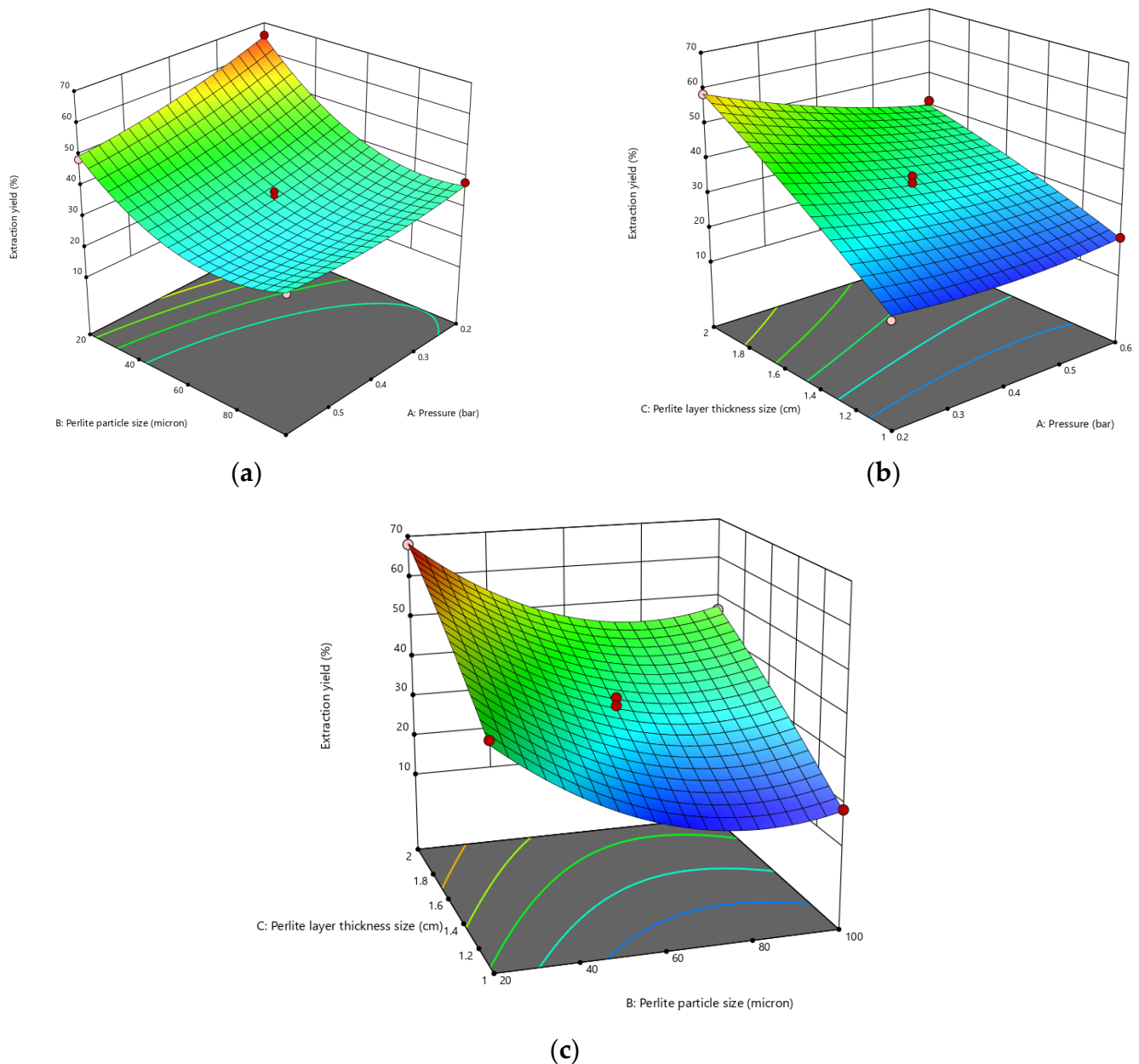


Figure 9. The effect of different variables on the extraction yield (a). Reaction of pressure and perlite particle size (b). Reaction of pressure and perlite layer thickness size (c). Reaction of perlite particle size and perlite layer thickness size.

3.7. Determining the Minimum Points in the Evaluation Range of the Filtration Process Using the Vacuum Filter System

To optimize the filtration process using the vacuum filtration system, energy consumption should be minimized, and the dependent variables should reach their maximum value. For this purpose, according to Table 5, the boundary conditions of the independent variables and the objective were determined. One of the important parts of optimization is weighting the objective function variables. Considering the equal importance of in-

dependent variables, a weight of 1 was assigned to them. The optimal conditions occur when the maximum separation efficiency and flow rate are achieved with the minimum energy consumption.

The optimal point for the filtration and separation process of pectin is shown in Table 6. The optimal conditions were obtained using the RSM method by computer at a vacuum level of 4/0 bar, perlite size of 60 microns, and a perlite layer thickness of 2 cm.

Table 6. Optimum level of independent variables for vacuum membrane filtering system.

Number	Pressure (bar)	Perlite Particle Size (micron)	Perlite Layer Thickness (cm)	Energy Consumption (Wh)	Flow Rate (mL/s)	Extraction Yield (%)
1	0.379	56.174	2	0.498	5.865	50.216
2	0.380	55.798	1.999	0.499	5.864	50.257
3	0.380	55.857	1.999	0.499	5.865	50.240
4	0.383	55.188	1.995	0.500	5.864	50.220
5	0.392	53.352	1.983	0.503	5.864	50.052

At the end, the optimal point proposed by the computer was evaluated experimentally in three repetitions, and the average of the dependent variables obtained was close to the empirical equation despite the sources of error. The recorded values, by applying the optimal conditions, were energy consumption of 54.0 Wh, a flow rate of 3.5 milliliters per second, and a separation efficiency of 2.54%. The values obtained by the empirical equation show an acceptable error rate (8%) compared to the values predicted by the model (row 1 of Table 6), indicating the correct choice of the model and its suitable solution for the data.

4. Conclusions

Considering the importance of pectin in various industries, a vacuum filtration system was developed for pectin separation from liquid and was studied in this research. The independent variables selected based on previous research were vacuum level, perlite particle size, and perlite layer thickness. The effects of these variables were investigated on energy consumption, separation efficiency, and flow rate as dependent variables. The results of the study can be summarized as follows:

1. A vacuum filtration membrane system can be used as an effective separation method in the pectin production process.
2. The dependent variable, separation yield, indicates the purity of the separated fluid, and the evaluation showed that the vacuum level, perlite particle size, and thickness of the perlite layer have an effect on its changes. Increasing the vacuum level leads to more impurities being sucked into the fluid and decreases the separation yield from 41% to 30%. Increasing the particle size from 20 microns to 60 microns decreases the yield from 55% to 33%, but increasing the particle size from 60 microns to 100 microns has no significant effect on the separation yield. The thickness of the perlite layer has the most significant effect on the separation yield, and by increasing it from 1 to 2 cm, the yield increased by 2.5 times. The maximum separation yield was achieved at a vacuum level of 0.2 bar, a particle size of 20 microns, and a thickness of 2 cm.
3. The level of vacuum and the size of the perlite particles affect the effective fluid flow changes. Increasing the vacuum level from 2.0 bar to 4.0 bar results in a 5.6 times increase in flow rate. However, with further increases in vacuum, the flow rate decreases. This trend is also observed for the size of perlite particles, indicating filter clogging and reduced flow rate at a vacuum level of 6.0 bar and perlite size of 100 microns.
4. Evaluation of energy consumption of the filtration system showed that the effective variables on energy consumption are the vacuum level and the size of perlite particles. With an increase in vacuum level from 2.0 bar to 6.0 bar, the energy consumption decreased by 5 times. The energy consumption for perlite size of 60 microns was

optimized to be 74.0 Wh, and coarser or finer perlite sizes had 5.4 times higher energy consumption.

5. The optimal conditions were obtained by the RSM method using a computer at a vacuum level of 4.0 bar, perlite size of 60 microns, and perlite layer thickness of 2 cm.

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