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# A Survey of Effective Parameters in Biomass Separation Using Vacuum Membrane Filtering: A Case Study of Pectin Acidic Solution <sup>†</sup>

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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 membrane filtering system for separation of fibrous materials from an acidic pectin solution under vacuum. These factors which include: filter-aid-particle size, amount of filter-aid (perlite) added to the solution, and the vacuum level, affect, separation quality, volumetric flow rate, and energy consumption. A vacuum separation device was developed for this purpose in order to separate the fibrous material dissolved in solution. The independent variables were examined at three levels, the data were analyzed, and 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 folds, while, further increase in the vacuum level decreases the flow-rate. This indicates clogging of the paper filter and decreased flow-rate at vacuum level of 0.6 bar and perlite particle size of 100 microns. The evaluation results showed that thickness of the perlite layer has the greatest effect on the separation efficiency and when increased from 1 to 2 cm, increases the efficiency 2.5 folds. The maximum value of separation efficiency was obtained at a vacuum level of 0.2 bar, particle size of 20 microns and perlite thickness of 2 cm. The energy consumption of 60-micron perlite was 0.74 Wh in the optimal state, and 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.

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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 get there, a driving force like 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, when the liquid approaches the filter medium, solid particles settle on the surface of the filter as a cake, while the liquid passes through the medium due to the negative pressure created by the vacuum. As the initial layer of cake is formed by perlite, the cake structure acts as a filter medium, and more solid particles are deposited on it and increase its thickness as the liquid passes through. Large industrial processes require 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.

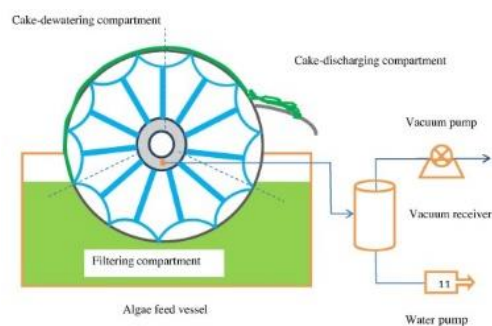


Figure 1. Rotary vacuum drum filter schematic [6].

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 [7]. The rotary vacuum drum filter is used in the pharmaceutical industry to collect calcium carbonate, magnesium carbonate, 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 [8]. Common problems with vacuum filters include premature clogging and settling of solid materials in the fluid storage tank [9]. Many engineering variables are playing 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 particles size, and the vacuum pressure required amount for filtration [8].

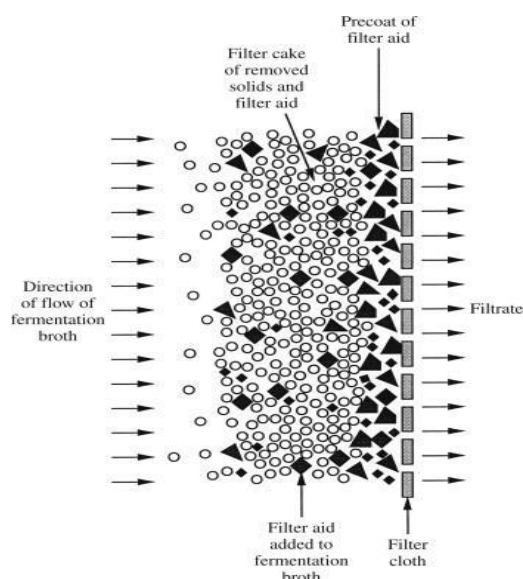


Figure 2. How Filter Assistance Operates [10].

Pectin is an acidic hydrocolloid compound which has many applications as a natural additive in various industries, especially in food, pharmaceuticals, and medicine [11]. Food industries, use it for its gelling, stabilizing, texture-creating, emulsifying, thickening, and fat-replacing features [12]. Additionally, in medicine and pharmaceuticals, it is used as a dietary fiber for the digestive treatment problems. The most common use of pectin in the food industry is in the jams production and jellies as a gelling and thickening agent [13], [14]. 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 [15]. Regarding its medical applications, pectin is really useful in preventing colon cancer and reducing blood cholesterol levels [16]. There are various methods for Pectin extraction.

The industrial method for pectin extraction is the extraction with acidic solutions at high temperatures. Traditionally, pectin is extracted by continuously stirring it in an acid solution at 80 to 100 degrees Celsius temperature for one hour. Pectin extraction depends on various factors, such as temperature, pH level, solvent properties, solid-to-solvent ratio, dried solid materials, particle size, and diffusion rate [17].

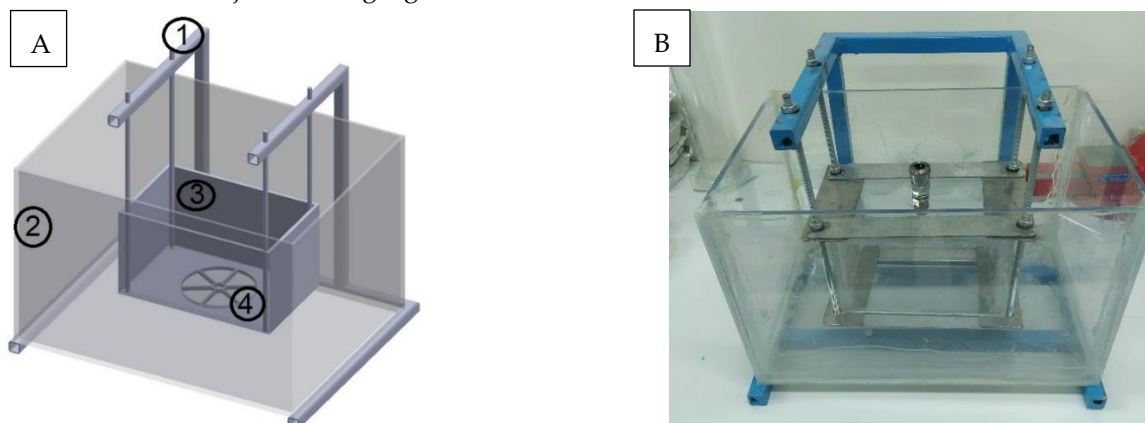
Given the mentioned applications of pectin, the increasing importance of productivity improvement in the food industry, especially in the field of recycling crucial materials such as pectin extraction, and the lack of scientific reports on the use of vacuum filters in separating such fluids have been the motivation behind this research. On an industrial scale, solid-liquid separation is performed using a combination of vacuum and membrane filters. On the other hand, regarding the important variables in vacuum separation, which have a significant impact on the quality and efficiency of separation and the fluid impurities are highly dependent on them, 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 include the size of the filter aid particles, the thickness of the filter aid layer, and the vacuum level of the filter, all of which have an effect on the dependent research variables, namely energy consumption, production yield, flow rate, and separation quality.

## 2. Method and materials

### 2.1. Design and construction method of a vacuum filter system

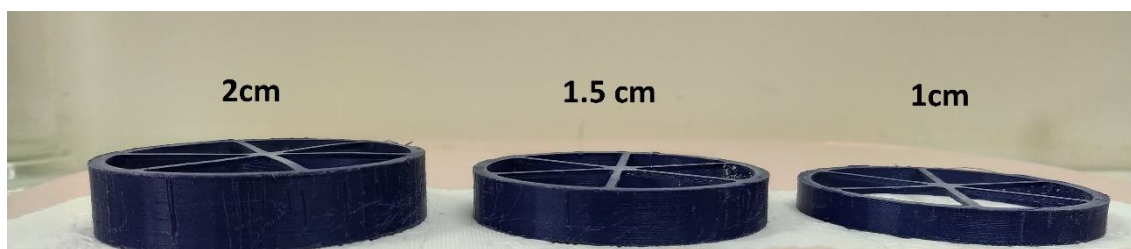
In order to simplify the components and apply dependent variable surfaces, a small-scale experimental vacuum filter system was constructed as described below. The trend chart of the rotary vacuum filter system is visible in Figure 3, which is explained further

below. In order to provide vacuum levels, a vacuum pump was used, which can be adjusted using a gate valve at those vacuum levels.



**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.

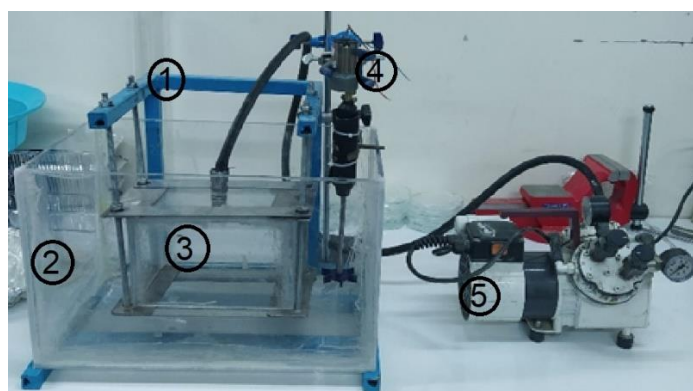
In order to utilize different sizes of filter aids (perlite) for separation, perlite with a specific size was used at three levels less than 20 microns, 40 to 60 microns, and 60 to 80 microns. Also, the variable thickness of the layer was considered at three levels of 1, 1.5, and 2 centimeters. Templates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



**Figure 4.** shows a view of 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 2 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, 5: and vacuum pump are listed in that order

### 2.2. Method of preparing filtration fluid

To prepare orange peel powder, 40 kilograms of fresh Thomson oranges were obtained from the fruit and vegetable market in Tehran, and their peels were separated for drying. The orange peels were dried in an electric oven at a temperature of 45 degrees Celsius for 24 hours. The dried peels were ground using a mill and sieved with a mesh number 40 sieve to ensure uniform particle size. Finally, the prepared powder was stored in a refrigerator at a temperature of 4- degrees Celsius for extraction. The orange powder was mixed with distilled water in a ratio of 1 to 25 and the pH of the solution was adjusted to 7.1 using hydrochloric acid. The solution was mixed using a mixer for 50 minutes at a temperature between 80 to 82 degrees Celsius (Figure 6). After the solution was prepared, it was passed through a cloth filter and cooled at room temperature. The resulting liquid is the substance tested in this study. In the standard laboratory process, centrifugation is performed to separate the pulp from the liquid, followed by the extraction of pectin from the liquid using ethanol in a ratio of 1 to 3. In this study, the separation stage is performed by centrifugation and filtration under vacuum.



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

### 2.3. Method of filtration using a vacuum filtration system

The filtration process using a vacuum filtration system In the first step, a specific volume of distilled water and perlite was poured into the tank to prepare the filter aid

layer. When the filter aid reached the desired thickness, the pump was turned off and the thickness of the perlite layer was adjusted. A three-dimensional structure made using three-dimensional coating technology was used to adjust the layer thickness. In the next step, a certain amount of prepared fluid was poured into the tank with a specific thickness of perlite, and by determining the desired vacuum level, the vacuum pump started to work and the mentioned solution passed through the filter aid and paper filter, and the separation time was recorded. In the final step, the filtered solution was diluted with ethanol (96%) at a ratio of 1:3 and kept at 4°C for 12 hours. Then, the pectin in the mixture of water and alcohol, which was floating in the form of clouds, was separated using a centrifuge at a speed of 10,000 revolutions per minute for 20 minutes. Finally, the obtained pectin was dried in an electric oven for 6 hours. In this study, the effect of vacuum levels (6.0 bar, 4.0, 2.0), perlite layer thickness (2 cm, 1.5, 1), and perlite particle size (100 microns, 60, 20) on energy consumption, flow rate, separation efficiency, and pectin extraction efficiency were evaluated.

#### 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. 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 passing through equation (1) was easily calculated.

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

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 hours 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 size (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 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 results of the analysis of variance (ANOVA) for energy consumption data by vacuum filtration system are presented in Table 2. The findings of this study indicate that the model is significant and the lack of fit is not significant. Therefore, the chosen model and analyses are reliable and valid. The effects of vacuum level and particle size second power at the 1% significance level and the effects of layer thickness of perlite and vacuum level second power and layer thickness of perlite at the 5% probability level are significant. The significance of the mentioned effects indicates the importance and influence of the selected independent variables in this experiment. The effects of vacuum level (A), particle size of perlite (B), and layer thickness of perlite (C) are shown in Table 2.

**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 <sup>a</sup>	0.0001
A-pressure	1	0.8902	0.8902	71.72 <sup>b</sup>	0.0001<
B- perlite particle size	1	0.0701	0.0701	<sup>a</sup> 5.65	0.0491
C- Perlite layer thickness size	1	0.0714	0.0714	1.46 <sup>a</sup>	0.0476
B × A	1	0.0181	0.0181	<sup>ns</sup> 1.08	0.2668
C × A	1	0.0004	0.0004	<sup>ns</sup> 0.0327	0.8615
C × B	1	0.0169	0.0169	<sup>ns</sup> 1.46	0.2820
A × A	1	0.4919	0.4919	<sup>a</sup> 39.63	0.0004
B × B	1	1.33	1.33	106.96 <sup>b</sup>	0.0001<
C × C	1	0.0789	0.0789	<sup>a</sup> 6.35	0.0398
residual	7	0.0869	0.0124		
Lack of fit	3	0.0706	0.0235	<sup>ns</sup> 5.77	0.0617
Pure error	4	0.0163	0.0041		
Cor total	16	3.1			

<sup>a</sup>significant at the 5% probability level.

<sup>b</sup>significant at the 1% probability level.

<sup>ns</sup> not significant.

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).

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

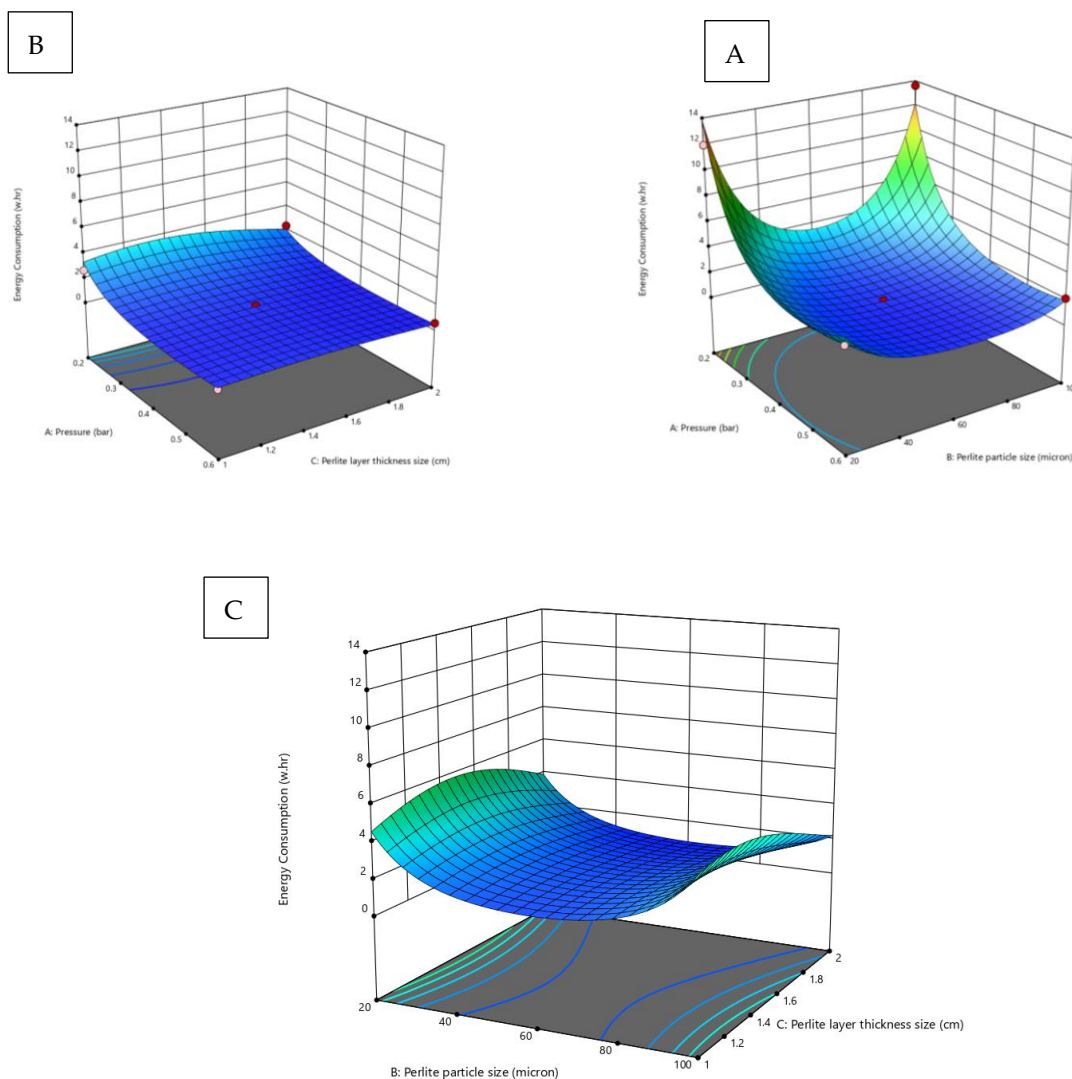
The variable E represents energy consumption in this equation, and based on the results of the regression model, the proposed model for predicting energy consumption with the selected independent variables is significant. The positive sign of each term indicates a synergistic effect, and the negative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

### 3.2. The interaction effect of independent variables on energy consumption

In Figure 7, the surface response charts of the dependent variable (energy consumption) are shown as contour lines for changes in the independent variables. Based on Figure 7a, it can be observed that the minimum energy consumption is evaluated at the vacuum level (bar 0.4). At the minimum vacuum level, the trend of changes in energy consumption is somewhat proportional to the increase in the size of perlite particles, and the minimum energy consumption value is obtained for particle size of 60 microns. At the maximum and minimum levels of perlite particle size, with an increase in vacuum level, energy consumption decreases significantly and then the trend of changes becomes less pronounced. It is evident that with an increase in vacuum level, the fluid passage time (assuming a constant volume) decreases, which is why energy consumption follows a decreasing trend. On the other hand, at the intermediate level of perlite particle size and minimum vacuum level, the filtration process has been performed well since the particle size is appropriate, and the filter has not clogged. Therefore, the energy consumption level in this case is less than other cases. Also, gradually with an increase in the thickness of the perlite layer at the minimum vacuum level, energy consumption has increased slightly and then decreased (Figure 7b).

The reduction in energy consumption in this state indicates that in thinner layers of perlite, considering that the auxiliary filter absorbs less solid fluid material, the filter is clogged faster and the filtration time increases, which will increase energy consumption. This process has helped filtration by increasing the thickness of the auxiliary filter with more solid material absorption, and the filtration process has been reduced, resulting in a downward trend in energy consumption. On the other hand, increasing the vacuum level reduces the filtration time, ultimately leading to a reduction in energy consumption. In other states, the trend of changes is insignificant. As shown in Figure 7-j, generally, the trend of changes in energy consumption is not significant. However, it should be noted that the trend of changes in energy consumption is partly proportional to changes in the size of perlite particles. When using perlite with a size of 20 microns, solid particles are difficult to pass through the auxiliary filter and increase the filtration process time. This is when using perlite with a size of 100 microns due to the easier passage of solid fluid particles through the auxiliary filter, which causes the filter to clog faster, and again increases the filtration process time and energy consumption. Manu Huttunen et al. [18], in a study to investigate energy consumption in a vacuum filter system by examining vacuum levels of 6.0, 4.0, and 2.0 bar, showed that changes in vacuum levels do not have a significant effect on energy consumption, and optimal energy consumption occurs at vacuum levels of 2.0 to 3.0 bar.





**Figure 7.** the effect of different variables on the energy consumption 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.

### 3.3. ANOVA results for fluid flow rate

The fluid permeability is equal to a specific volume of fluid passing through the filter section per unit of time. The results of ANOVA for the effect of different variables on the production rate are presented in Table 3. The ANOVA results showed that the model is significant and the lack of fit is not significant. Therefore, the chosen model and analyses are reliable and valid. The effects of the vacuum level and vacuum level squared, perlite particle size, and perlite layer thickness are significant at a 5% probability level. The significance of these effects indicates the importance and impact of the selected independent variables in this experiment. The effects of vacuum level (A), perlite particle size (B), and perlite layer thickness (C) are shown 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 <sup>a</sup>	0.0005
A-pressure	1	0.9039	0.9039	47.55 <sup>a</sup>	0.0002

B-perlite particle size	1	0.0196	0.0196	ns 1.03	0.3442
C- Perlite layer thickness size	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	<sup>a</sup> 37.17	0.0005
B × B	1	1.22	1.22	<sup>b</sup> 64.03	0.0001<
C × C	1	0.1203	0.1203	<sup>a</sup> 6.33	0.0401
residual	7	0.1331	0.019		
Lack of fit	3	0.1103	0.0368	6.45 <sup>ns</sup>	0.0518
Pure error	4	0.0228	0.0057		
Cor total	16	3.21			

<sup>a</sup>significant at the 5% probability level.

<sup>b</sup>significant at the 1%probability level.

<sup>ns</sup> 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).

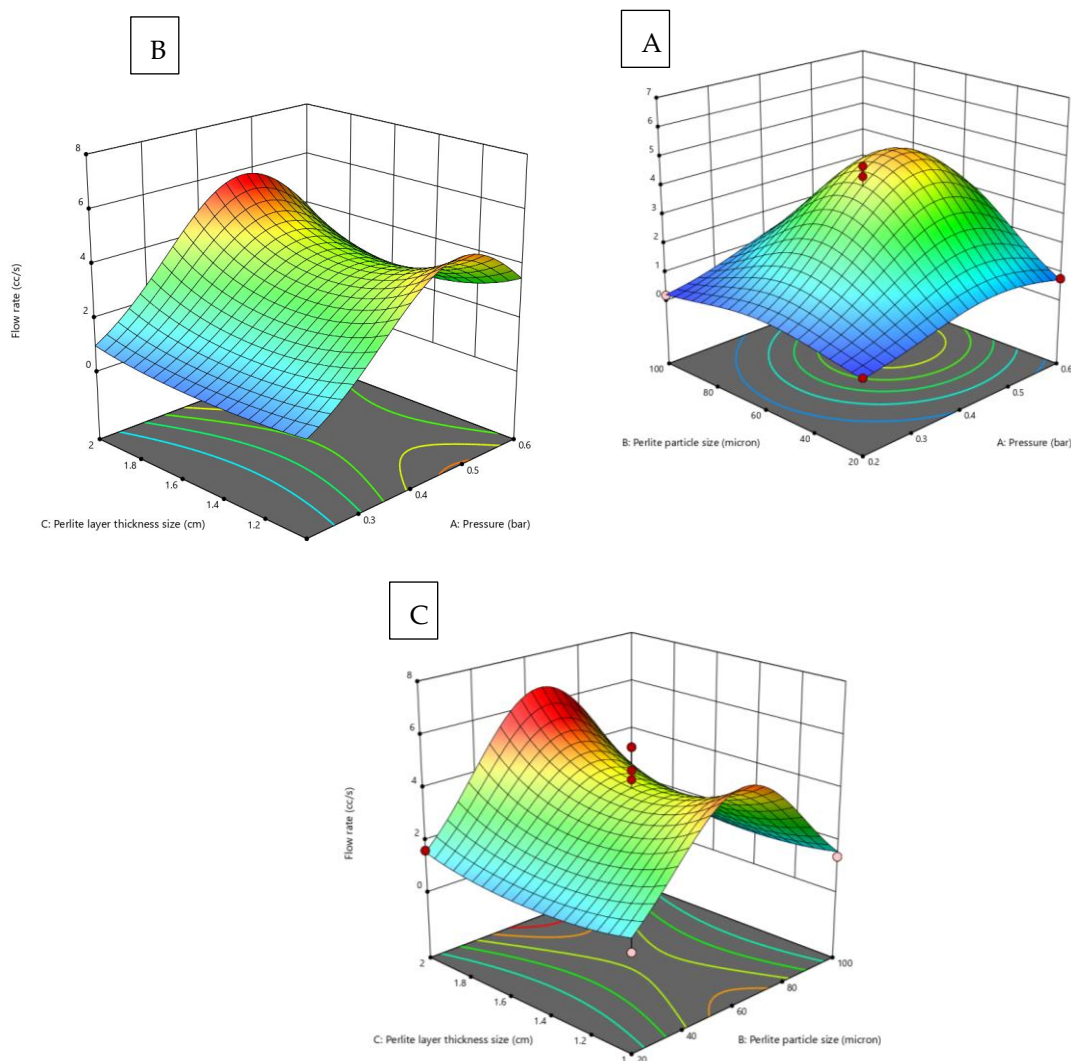
$$\text{Log}_{10}^q = -1.28 + 9.51 * A + 0.03 * B - 2.04 * C + 0.009 * A * B - 0.46 * A * C + 0.002 * B * C - 10.38 * A^2 + 0.0003 * B^2 + 0.68 * C^2 \tag{4}$$

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, with an increase in the size of perlite particles, the changes in the flow rate are proportionate, and this trend is more intense with an increase in the vacuum level. Similarly, with a fixed size of perlite particles, the trend of changes in the dependent variable is proportionate to an increase in the vacuum level. The maximum value of volumetric flow rate (fluid passing through the filter section) was obtained 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 in the lower level. Considering the reasons mentioned, the intermediate size of perlite particles is suitable, and on the other hand, the trend of changes in the vacuum level on the dependent variable shows that at maximum vacuum level, with a faster flow of fluid, the possibility of clogging has increased both in the auxiliary filter and in the filter section. Therefore, a decrease in the flow rate passing through the filter section has been 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 changes in the dependent variable is slight, but with an increase in the vacuum level, a partially increasing trend was observed. On the other hand, considering a specific thickness of the support filter, with an increase in the vacuum level, the

increasing-decreasing trend of the dependent variable is 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<sup>2</sup>) 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 has decreased, so the maximum particle size of the perlite layer is desired. As shown in Figure 8j, 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 has increased, and the filtration process has not been well performed, and the dependent variable has decreased. In maximum and minimum particle sizes of perlite, the trend of changes in the dependent variable is negligible with an increase in the thickness of the perlite layer. On the other hand, 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.



**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 size C. reaction of perlite particle size and perlite layer thickness size.

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 <sup>b</sup>	0.0001<
A-pressure	1	168.54	168.54	60.19 <sup>a</sup>	0.0001
B-perlite particle size	1	940.48	940.48	335.89 <sup>b</sup>	0.0001<
C- Perlite layer thickness size	1	1609.71	1609.71	<sup>b</sup> 574.9	0.0001<
B × A	1	41.54	41.54	<sup>a</sup> 14.84	0.0063
C × A	1	82.54	82.54	<sup>a</sup> 29.48	0.0010
C × B	1	0.8742	0.8742	<sup>ns</sup> 0.3122	0.5937
A × A	1	16.08	16.08	<sup>a</sup> 6.00	0.0442
B × B	1	460.44	460.44	<sup>b</sup> 164.44	0.0001<
C × C	1	5.36	5.36	<sup>ns</sup> 1.91	0.2092
Residual	7	19.6	2.8		
Lack of fit	3	8.17	2.72	0.9531 <sup>ns</sup>	49.54
Pure error	4	11.43	2.86		
Cor total	16	3351.93			

<sup>a</sup>significant at the 5% probability level.

<sup>b</sup>significant at the 1%probability level.

<sup>ns</sup> not significant

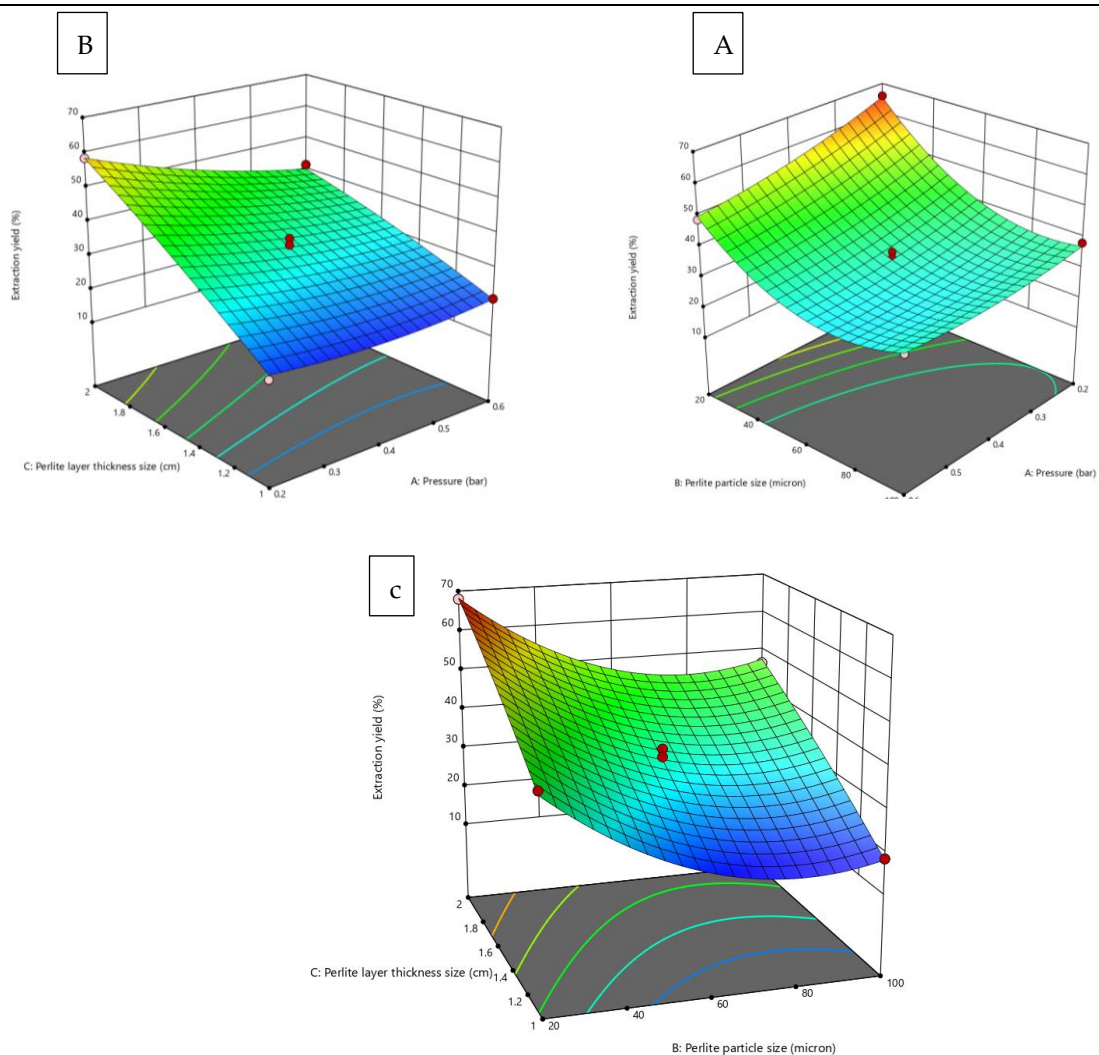
Using the response surface method, a full second-degree polynomial model with a determination coefficient of 42/99 was selected to estimate the value of separation efficiency by varying the levels of independent variables in the coded form. The proposed actual model to examine the effects of selected variables is a second-degree polynomial function with the equation (5). (42/99 = R2)

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

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 aid filter, so the trend of variable-dependent separation efficiency decreases. 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 9j shows, with an increase in the thickness of the aid filter 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 changes with a specified thickness of the aid filter layer is also bilinear. Although the maximum separation efficiency is desirable, it is achieved at the minimum size of perlite particles (20 microns) and the 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. Also, Davarzani et al. [21] 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. Additionally, Filadieu et al. [22] 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, the 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 independent 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.

**Table 5.** independent and objective programs 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 size	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

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 micron, 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 size (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 proposed optimal point 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 predicted values by the model (row 1 of table 6), indicating the correct choice of the model and its suitable solution for the data.

#### 4. Conclusion

"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 on energy consumption, separation efficiency, and flow rate were investigated 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 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 centimeters, 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 centimeters.

3- The level of vacuum and the size of the perlite particles affect the effective fluid flow changes. With an increase in vacuum level from 2/0 bar to 4/0 bar, the flow rate has increased by 5/6 times, but with further increase in vacuum, the flow rate decreases. This trend has also been 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 has 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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