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Original research

Kinetic modeling of pectin extraction by ultrasound assisted and conventional methods

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A B S T R A C T

The goal of this study was to investigate the most suitable kinetic models for pectin extraction by way of ultrasound-assisted extraction (UAE) and conventional heating extraction (CHE) with acid from melon peel. The ultrasound and acidic assisted extraction were performed on powdered melon peel in three different temperatures of 40-55-70°C and 50-70-90°C, respectively. It was observed that, in the UAE method, pectin yield increased significantly in a shorter time compared to CHE which reached its peak at a lower temperature so UAE can be considered as more reasonable method. The experimental data (pectin yield versus time) were suited to a variety of kinetic models by linear regression. The total extract yields from melon peel by UAE and CHE among five conditions displayed the closest fit in the condition of pH=2 at temp 55°C to the power law model (R^2 = 0.92) and in the condition of pH = 2 at temp 70°C to the parabolic diffusion model (R^2 = 0.94), respectively. The theoretical models expressed the extractability, dissolution and degradation rates of pectin, and investigated the extraction kinetics. When ultrasound and acidic procedures were applied simultaneously a synergetic effect between heating and extractability, dissolution and degradation of pectin was observed leading to a higher yield (34.18%), extractable pectin (37.45%), degradation rate (0.23%), dissolution rate (5.37%), with a shorter extraction time (61.28 min).

Keywords: Ultrasound assisted extraction, Acidic extraction, Kinetic models, Degradation, Dissolution rate

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1. Introduction

Melon peel can be an outstanding source of pectin. It is related to the family *Cucumis melo* L. Iran is one of the main producers of melons throughout the world (Barzegar et al., 2013). Some portion of the fruit peel waste could be consumed as animal feed, but the majority of the processing waste is disposed and consequently can cause environmental pollution. Hence, the disposal of melon peel waste has strong potential for the development of valuable ingredients and mitigation of environmental hazards. Accordingly, consuming by products have been used for alternative applications, specially producing natural compounds including pectin, flavonoids, carotenoids, limonene and polymethoxy flavones. Pectin belongs to a family of complex hetero polysaccharides which consist of linear polymers of D-α-(1→4) galacturonic acid (Vidal, 2001). Pectin has different applications as thickening and gelling in jams, soft drinks, and dairy products (Jitpukdeebodintra et al., 2009). Conventionally, pectin is extracted in a hot dilute mineral and organic acidic solution (Zhongdong et al., 2006). The time required depends on some factors, such as the particle size and shape of raw material (Yuting et al., 2014) and the type of pectin desired, and the condition of extraction; however, generally this process can be considered as a time consuming process (May, 1990). Overall, among different methods of extraction conventional heating extraction (CHE) has both quantitative and qualitative disadvantages for pectin extraction (Li et al., 2006). Due to adverse effects associated with the CHE method (Faravash & Ashtiani, 2008), using alternative methods could achieve better quality and quantity of extracted pectin. With the purpose of reducing the extraction time and improving the extraction yield, new techniques need to be promoted. Ultrasound assisted extraction (UAE) is the most technical of the industrially used methods, promoting mass transfer phenomena (Corrales et al., 2008). Ultrasound has been developed for potential application in the extraction of proteins, oils and bioactive compounds from plants, specially polyphenols, anthocyanins, pectin, tartaric acid, aroma compounds, polysaccharides and functional compound material (Vilkhu et al.,

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2008). Nowadays, using pulsating hydrodynamic action with turbulent recirculation (Kratchanov et al., 1986) leads to increase yield, reduced process time and production cost. Hence, the aims of this work were to determine the kinetic parameters that describe the mechanism of pectin extraction by UAE and CHE in order to study the effects of various parameters on the yield and kinetics of extraction. A substantial number of kinetic models have been implemented for solid–solvent extraction due to the hypothesized and simplified extraction mechanism and utilized in the extraction of food and medicinal products from water and organic solvents (Kitanovic et al., 2008; Meziane & Kadi, 2008; Panchev et al., 1989). Panchev (1989) presented a model to describe the kinetics of pectin extraction from apple pomace, involving the dissolution of pectin from protopectin and the degradation of the dissolved pectin. Furthermore, (Cheung et al., 2013) evaluated the suitable kinetic models for ultrasound-assisted extraction of water-soluble polysaccharides from the fruit body and medicinal fungi. These model equations are practical for design and optimization of the extraction processes and for analyzing and detecting the major factors and their effects over extraction. The suitable models or the kinetic characteristics depend on the final materials which are being extracted. To the best of our knowledge, no previous studies have been revealed on kinetic models for UAE and CHE for extraction of pectin from by-products. The goal of this study was to evaluate the suitable kinetic models for pectin extraction by UAE and CHE, to achieve a better understanding of which parameters affected the extraction rate in UAE and CHE; in addition, various comparisons were also conducted between the mentioned procedures.

2. Material and Methods

2.1. Preparation of materials

The collected melon peels were pretreated with minor modifications. At first the samples were passed through a 60-mesh sieve to attain particles < 300 µm, after that they were soaked in a water bath at 90°C for 15 min to lead inactive enzymes, then dried in an oven with air circulation at 50°C. In the final stage, samples were vacuum-packed in glassware and stored until use (Hosseini et al., 2016).

2.2. Pectin extraction from melon peel by CHE

To extract the pectin with CHE, 5 g of powdered melon peel was mixed with 100 ml acid solution (1:20 solid/liquid) by citric acid (99%) which was purchased from Merck Chemical Co (Darmstadt, Germany) to adjust the pH level and pectin was extracted based on the extraction conditions established by the experimental design. A water bath heating was used at (50, 70, 90°C) and the extraction was performed in time (20, 50, 80, 110, 140 min) and pH (1.5, 2, 2.5) (Luthria, 2008). Afterwards, the hot extract was filtered using Whatman paper No.1 and the filtrate was centrifuged at 5000 rpm for 30 min to achieve higher purifications of pectin in the solution. Then the supernatant was precipitated with 96 % ethanol (1:2 % v/v). After storing the solution for 24 h at 4 ˚C, the polysaccharides were washed two times with ethanol. Finally, the precipitated polysaccharides were dried under vacuum (ACE400L, ATRA, Tehran, Iran) at 50°C to a constant weight (Sahari et al., 2003).

2.3. Extraction of pectin using UAE

Single-factor experiments in UAE (UP200H, Hielscher Ultrasonics, Germany) were employed to determine the effects of different parameters including temperature (40, 55, 70°C), sonication time (10, 25, 40, 55, 70 min), pH (1.5, 2, 2.5) with duty cycle 50% , ultrasound power of 70 W and frequency of 24 kHz on pectin yield according to the preliminary experiments (Bagherian et al., 2011). After extraction, the extraction mixture was centrifuged at 6000 rpm for 30 min under 4°C using a low-temperature centrifuge. Then the supernatant was precipitated with 96% ethanol (1:2 v/v). After storing the solution for 24 h at 4°C, the polysaccharides were washed two times with ethanol. Finally, the precipitated polysaccharides were dried under vacuum at 50°C until its weight was constant.

The yield of extraction was calculated according to the following formula:

$$\text{yeild (\%)} = \frac{\text{weight of extracted pectin}}{\text{weight of dried melon peel}} \times 100 \quad (1)$$

2.4. Physicochemical properties

Crude protein (N × 6.25) and total ash content of extracted pectin in both methods were measured according the methods which are published by (Raji et al., 2017) (Table 1).

Table 1. Physicochemical properties comparison between UAE and conventional methods.

Extraction method	Protein (%)	Ash (%)
CHE	3.1	3.5-4
UAE	4.1	2.5

2.5. Extraction kinetics study

2.5.1. Kinetic models

Six empirical kinetic models for solid-liquid extraction were applied to fit the experimental data from the UAE and CHE of melon peel extraction as revealed in Table 2 and Table 3. These models have been previously applied to the extraction of natural products from various sources such as food and medicinal plants (Velickovic et al., 2006; Kitanovic et al., 2008).

In this study, dried pulps were milled and the obtained powder was sieved (50-mesh size)

The diffusion coefficient of the extractable component is constant. Solid particles are well dispersed in the extracting solvent, additional assumptions are also applied for different models. Both the unsteady diffusion model and the parabolic diffusion model assume a two-step extraction mechanism, the initial and rapid washing step for the compounds on the particle surface, followed by diffusion through the particle. The power law model describes the extraction mechanism by the diffusion of compounds through a non-swelling device. The Elovich's equation, which is widely applied to chemisorptions (McLintock, 1967) signifies that the extraction rate of a substance from solid declines exponentially with the extraction yield ($\frac{dy}{dt} = \beta \exp^{-\alpha y}$). (Kitanović et al. 2008) found that the extraction data of St. John's Wort with boiling ethanol-water could be fitted well to five kinetic models and the best to Elovich's equation (Kim et al., 2002) that successfully applied the hyperbolic model for the extraction of nuclides from

paraffin waste and total polyphenols from grape seeds, respectively (Bucić-Kojić et al., 2007).

All experiments were performed in triplicate, The experimental data, total extract yield or polysaccharide yield from the water extract versus time of UAE and acidic extraction for melon peel powder were fitted to the kinetic models by linear regression analysis to derive the model parameters. The goodness of model fit to the experimental data was evaluated by the correlation coefficient (R^2) together with the statistical significance indexes, F and the relative p value.

Model equations and constants were as below:

1- Parabolic diff: $Y= y0+Dt^{0.5}$ y0=initial yield D=Diffusion coeff

2- Power law: $Y= B*t^n$ B=rate constant n=diffusional exponent

3- Weibull: $Y=1-e^{(-t_m/D)}$ D= scale parameter m= shape factor

4- Elovich: $Y= E_0 +E_1 Lnt$ E_0=initial yield E_1 Lnt=initial extraction yield

5- Unsteady diff: $Y=(1-b)e^{-kt}$ b=Washing coef k=rate constant

6- Peleg: $Y=c_1*t/(1+c_2*t)$ c_1=initial rate c_1/ c_2= capacity constant

2.5.2. *Kinetic study*

Mathematical models develop theoretically, so that the process of pectin extraction from melon peel concludes two simultaneous transformations: The insoluble pectin turns into soluble pectin and diffusion of the pectin from plant tissues into the solution describe with rate constant k_1, and the partially dissolved pectin is degraded with rate constant k_2 (Panchev et al., 1989). In this study, the mathematical model for the extraction kinetics of pectin was described according to(Panchev et al., 1989).

The scheme for extraction of pectin is according to the below trend:

Protopectin → dissolved pectin → degraded pectin

The amount of protopectin that can be extracted from the material under some specified conditions after extraction for a time *t* was marked as *z(t)*, the amount of the degraded pectin as *q(t)*; and the dissolved pectin that will be obtained as *y(t)*. Then we can get the following first- order reaction kinetics:

$$\frac{dz(t)}{dt} = -k_1\, z(t) \quad (2)$$

$$\frac{dq(t)}{dt} = k_2\, y(t) \quad (3)$$

$$\frac{dy(t)}{dt} = k_1\, z(t) - k_2\, y(t) \quad (4)$$

P_E was used to express the percentage of the extractable pectin of melon peel under specified extraction conditions. Then,

$$P_E = z_t + q_t + y_t \quad (5)$$

The following equations indicate the changes with time of protopectin *z(t)*, the obtained pectin *y(t)*, the degraded pectin *q(t)*. The dissolved and obtained pectin that will be calculated with describing the change with time as $y_{(t)}$:

$$y(t) = P_E.K_1/(K_2 - K_1)(\exp(-k_1.t) - \exp(-k_2.t)) \quad (6)$$

We can point out the time t_{max}, at which the pectin yield reached its maximum value, y_{max}.

$$t_{max} = \frac{\ln\left(\frac{k_1}{k_2}\right)}{k_1 - k_2} \quad (7)$$

$$y_{max} = P_E.\frac{\left(\frac{k_2}{k_1}\right)^{\frac{k_2}{k_1}}}{1 - \frac{k_2}{k_1}} \quad (8)$$

The kinetics curves were suited for Eq. (6) and the maximum value of pectin yield y_{max} and the time t_{max} at which the pectin yield reached y_{max} were calculated from Eqs. (7) and (8) (Yuting et al., 2014).

3. Results and Discussion

3.1. *Investigation of pectin extraction by CHE*

The effects of extraction temperature on the extraction yield of pectin using the CHE method from five conditions have illustrated in Fig. 1 to maintain the best optimum time for extraction (Yapo et al., 2007). Fig. 1 showed that the pectin yield in the CHE method dramatically increased with increasing temperature, this increase was mainly due to the improvement of pectin solubility caused by temperature increase; however, thermal degradation of pectin at high temperature leads to the decrease of pectin yield(Panchev et al., 1989). In the Fig. 1 for most conditions, the extract yield increased throughout the entire period, but in hard conditions (over 110-140 min) illustrated a constant rate. The effect of temperature is illustrated in Fig. 1, while some experiments had the same pH, pectin extraction was significantly temperature dependent and mass transfer increased with more temperature, such that the pectin yield of temp 90°C was 10% followed by a temp of 70°C, which was approximately 8%, and at a temp of 50°C accounting for nearly 5%. Fig. 1 with the pH = 1.5 and temperature 70°C has shown different trends, in which the yield of extraction represented a steeper increment, compared to other conditions, to reach the peak at almost 59% and then in the later periods yield of extraction decreased dramatically to reach starting point which was 40%.

Table 2. correlation coefficients (R^2) and model constants for various kinetic models fitted by linear regression to the CHE method experimental data in five different conditions.

Model		pH=2.5 temp=70°C	pH=2 temp=90°C	pH=2 temp=70°C	pH=2 temp=50°C	pH=1.5 temp=70°C
	R^2	0.92	0.85	0.94	0.63	0.64
Parabolic diffusion	Y_0	-0.004	064	0.178	2.34	0.007
	D	0.067	0.0085	0.082	2.105	0494
	R^2	0.85	0.79	0.81	0.76	0.83
Power law	B	032	0.41	0.25	0.38	0.45
	n	0.024	0.031	0.021	0.034	0.036
	R^2	0.67	0.89	0.78	0.53	0.69
Weibull's exponential	D	2.67	2.46	1.87	1.45	4.21
	m	0.076	0.071	0.056	0.064	0.021
	R^2	0.78	0.79	0.93	0.71	0.86
Elovich's logarithmic	E_0	0.56	0.34	0.65	0.68	0.2
	E_1	0.034	0.025	0.038	0.015	0.016
Unsteady diffusion	R^2	0.56	0.65	0.41	0.58	0.71
Peleg hyperbolic	R^2	0.43	0.48	0.39	0.18	0.51

Table 3. Correlation coefficients (R^2) and model constants for various kinetic models fitted by linear regression to the UAE experimental data in five different conditions.

Model		pH=2.5 temp=55°C	pH=2 temp=70°C	pH=2 temp=55°C	pH=2 temp=40°C	pH=1.5 temp=55°C
	R^2	0.70	0.45	0.74	0.63	0.34
Parabolic diffusion	Y_0	0.0032	025	0.003	0.0402	0.26
	D	0.074	0.0075	0.20	0.006	0.005
	R^2	0.89	0.79	0.92	0.76	0.87
Power law	B	032	0.41	0.25	0.38	0.45
	n	0.024	0.031	0.021	0.034	0.036
	R^2	0.87	0.89	0.78	0.83	0.69
Weibull's exponential	D	3.35	4.46	1.87	3.45	4.21
	m	0.076	0.071	0.056	0.064	0.021
	R^2	0.91	0.89	0.83	0.79	0.86
Elovich's logarithmic	E_0	0.56	0.34	0.65	0.68	0.2
	E_1	0.034	0.025	0.038	0.015	0.016
Unsteady diffusion	R^2	0.56	0.65	0.71	0.58	0.61
Peleg hyperbolic	R^2	0.43	0.18	0.49	0.48	0.51

Table 4. Kinetic parameters of pectin extraction from melon peel.

Extraction method	Extractable pectin P_E	Dissolution rate constant k_1	Degradation rate constant k_2	Optimal extraction time t_{max}	Maximum yield y_{max}
CHE	37.45	5.37	0.23	61.28	34.18%
UAE	24.61	4.54	0.16	76.36	22.67%

3.2. Investigation of pectin extraction by UAE

In the UAE method results showed that pectin yield took a sharp upward trend in a shorter time and then declined with a rise in temperature. In some cases, the decrease of pectin yield could partially be as a result of the decrease in power output, since it has been showed that temperature noticeably affected the ultrasonic power output (Athanasia, 2013). In all conditions yield reached a peak and then slowed down gradually by contrast, at a pH = 1.5 and temperature = 55°C, yield of extraction after the peak in the graph at almost 28% reduced drastically with a difference of extraction amount. It is worth noting that, the most outstanding factor in the yield is pH since the lowest and highest proportion of yield over this time belonged to pH 2.5 and 1.5 which were (0.02-0.06) and (0.2 -0.4), respectively (Raji et al., 2017); also, the conditions with the pH 2 stood at the middle level.

3.3. Comparison between the CHE and UAE method

Fig. 2 showed that the yield of extraction in UAE reached the maximum level in a shorter time compared to the CHE method, which showed that UAE is more affordable. In terms of a comparison between the CHE and UAE method, it can be observed that the extract yield versus the time for the former method in over

conditions exhibited a linear increase in the early period and a linear decrease in the later period; whereas, the extract yield versus the time of the latter method showed a linear increase in the early period and remained constant in the later period. This loss at the later period over conditions could be rooted in the degradation of pectin when exposed to heating and UAE radiation, such that it was stronger in CHE method in comparison with UAE method (Koubala et al., 2008).

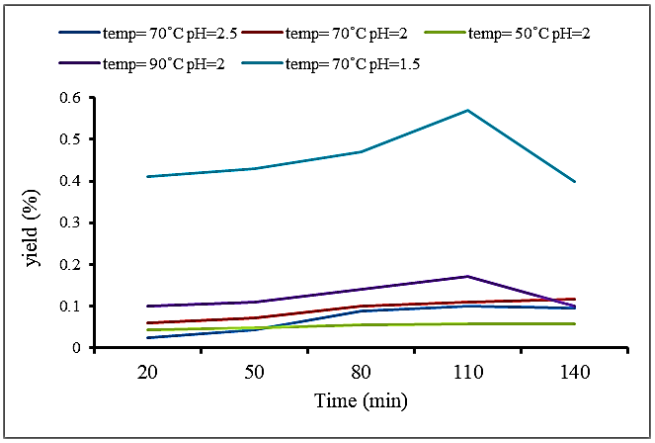

Fig.1. The change of total pectin extract yields (w/w) with time of conventional method in five conditions.

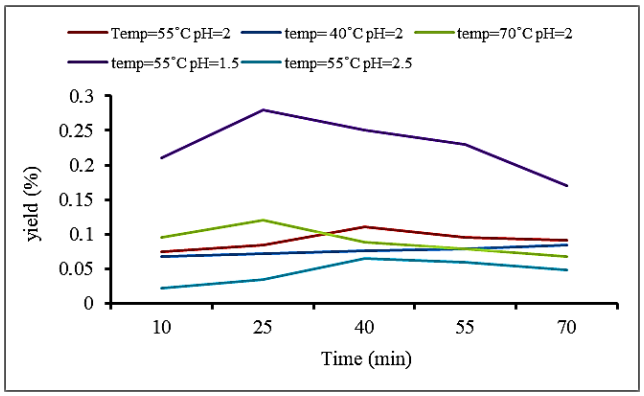

Fig. 2. The changes of total pectin extract yields (w/w) with time of UAE in five conditions.

3.4. Physicochemical features

Characteristics of melon peel including ash and protein were analyzed at a temperature of 70°C and pH 2 using CHE method and at a temperature of 55°C and pH 2 using UAE method (Table 1). The protein content of melon peel was 3.1% and 4.1% w/w in CHE and UAE methods, respectively. The most influential factor on protein content has been known to be temperature; as the temperature increases, the interactions between protein and pectin are destroyed (Yapo et al., 2007).

The ash content of pectin extracted under optimized conditions was between 3.5 and 4% w/w in the acidic method and 2.5% in UAE. Ash content rose with increasing acid strength and temperature to solubilize indigenous minerals in the peel. The solubilized mineral would then precipitate with pectin during alcohol precipitation (Sahari et al., 2003).

All of the mentioned reports illustrate that the pectin from UAE method was purer and more fruitful due to the better and softer condition.

3.5. Kinetic analysis for the CHE method

The Table 2 illustrates the correlation coefficients (R^2) from the linear regression fit of the experimental data (yield versus time) to different kinetic models for five conditions including (pH 2.5 at a temp of 70°C, pH 2 at 90°C, pH 2 at 70°C, pH 2 at 50°C, pH 1.5 at 70°C) and the corresponding model parameters in the CHE method. For the conventional method pH 2 and temp 70°C as the most practical condition showed were a close fit to the Elovich's logarithmic and Parabolic diffusion models with relatively large R^2 values 0.93 and 0.94, respectively. Due to the fact that, the most suitable models showed a close fit to the experimental data with a large R^2 values. Meanwhile, pH 2 at a temp of 70°C among five conditions represented a large F value (4896.14) and a small p value (< 0.01) with the mentioned models from regression which is implicated the significance of statistics in the models fit. The most suitable models were categorized in a two stage extraction process; an initial washing stage (to an initial yield y_0) followed by a slow stage (with the yield growing linearly with $t^{1/2}$).

Among the six models, the Peleg hyperbolic and unsteady diffusion models for most conditions attained the smallest R^2 values (< 0.71) and can be considered unsuitable for the CHE method kinetics with pectin of melon peel. Similarly, the smallest R^2 value (0.18) among kinetic models, over five conditions, belonged to the condition where the pH was 2 at a temp of 50°C in Peleg hyperbolic model. Indeed, among the five conditions, the one with a pH of 2 at a temp of 50°C and pH of 1.5 at a temp 70°C did not show a most suitable close fit with any of the models and represented a lower R^2 value compared to the other conditions. The significant differences of two mentioned conditions are specified by coefficients which had F value 1044 and 1740 respectively.

3.6. Kinetic analysis for UAE method

The given Table 3 illustrated the correlation coefficients are related to the linear regression fit of the experimental data to different kinetic models for all conditions as below: pH = 2.5 and temp = 55°C, pH = 2 and temp = 70°C, pH = 2 and temp=55°C, pH = 2 and temp = 40°C, pH=1.5 and temp = 55°C in the UAE method. In this method, the Power law, Weibull's exponent and Elovich models, were a close fit to the experimental data of five mentioned conditions with fairly great R^2 values (0.76-0.92), (0.69-0.89), (0.73-0.91), respectively and the parabolic diffusion, hyperbolic and unsteady diffusion models for most conditions attained the smallest R^2 values (< 0.74), and can be considered unsuitable for the UAE method kinetics with pectin from melon peel. It was clear that among all the models, the highest R^2 value (0.92) was obtained from the condition of pH 2 and temp 55°C in power law model, which should be considered as the most suitable and applicable model for pectin extraction using UAE method. The most fruitful condition with the highest F value (1618) was related to pH 2 and temp 55°C. These results are reasonable since, the condition with the moderate temperature and pH showed a much closer fit with the mentioned models. According to some research, the power law model is considered the most applicable model for

the extraction of a substance from a non-swelling device (Sinclair & Peppas, 1984). As can be seen during our experiments, the powder of melon peel in all conditions expressed as well-dispersed particles in the extracting solvent.

3.7. Heating effects on the kinetic extraction

The extractable pectin P_E rate constants k_1 and k_2, t_{max} and y_{max} for both methods at the optimal level were calculated and presented in Table 4. The results showed that rising temperature affected the growth of P_E, k_1 and k_2, which originates from proto-pectin hydrolysis and mass transfer. This was agreed with accepted conclusion, in which the hydrolysis of pectin is correlated with an increment in temperature (Bagherian et al., 2011).

Table 4 showed that P_E, k_1 and k_2 of the CHE method was much higher than that of UAE. The given table illustrated that the dissolution rate (k_1) caused by CHE, accounting for 5.37%, was much more than 4.54%, which was caused by UAE. P_E and k_2 showed the same trend too and according to Zhang et al. (2013), the results of UAE reported that the degradation efficiency was lower than CHE with the increase of temperature and the degradation of pectin, which was not fruitful, was higher in the CHE method. This indicated that with the addition of acid heating had stronger effects on the improvement of extractability and the dissolution rate of pectin to that of heating with irritation. The study of depolymerization of pectin in the solid-liquid mixture was difficult since the extraction system was too complicated (Panchev et al., 1989).

4. Conclusion

The studies of pectin extraction modeling from melon peel using the acidic Method and UAE with different conditions, reported that the kinetics of extraction was significantly dependent on some factors, especially temperature, pH, time and the type of extraction method. In comparison with the CHE method, ultrasound was effective in enhancing the extraction of pectin at a relatively low temperature and short time from melon peel to reach the maximum yield. The modified models including power law and parabolic diffusion were detected to predict the best design with higher quality and yield of pectin extraction. Meanwhile, the mentioned models described the extraction kinetics involving pectin extractability, dissolution and degradation rates. The maximal yield and optimal extraction time could be obtained by modeling the extraction process of different methods. Both CHE method and ultrasound method had crucial effect on the promotion of extractability, dissolution rate and degradation rate of pectin and there was a synergistic effect between heating temperature and time on the extraction of pectin. The results found that UAE could be an efficient and innovative technique for pectin extraction from fruit and plant materials for industrial application with perfect and purer pectin with lower degradation rate whereas, CHE leads to pectin degradation, so it could not be considered an appropriate method for the quality of the pectin extraction.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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