Food Research 8 (Suppl. 2) : 102 - 107 (2024)
Journal homepage: https://www.myfoodresearch.com

FOOD RESEARCH

RESEARCH PAPER

Evaluation of drying and extraction parameters for the extraction yield of watermelon (*Citrullus lanatus* (Thunb.)) rind using statistical design experiment

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Article history:
Received: 5 March 2023
Received in revised form: 27 September 2023
Accepted: 20 January 2024
Available Online: 6 April 2024

Keywords:
Watermelon rind,
OFAT,
Sonication extraction,
Oven drying,
Dehydrator drying

DOI:
https://doi.org/10.26656/fr.2017.8(S2).61

Abstract

Watermelon rind contributes about 30% of overall watermelon mass and is considered the most underutilized resource as it is usually discarded as waste. Watermelon rind resources have great potential economic value in various industries. Thus, utilizing the rind could decrease the amount of biological waste in the environment. Therefore, this study aims to determine the optimal conditions in extracting oven and dehydrator drying watermelon rind using a sonicator extraction procedure. Watermelon rind samples were evaluated using a one-factor-at-a-time (OFAT) evaluation to identify the most significant factor for the sonicator extraction time (0.5-3 hrs), ethanol concentration (20-100%), solvent-to-solid ratio (10:1-50:1 v/w), and sample drying time (24-48 hrs) parameters. The highest yield obtained for the oven drying sample was at one hr extraction time (15%), 100% ethanol concentration (12.5%), 30:1 v/w ratio (12%), and 48 hrs drying (9%). Meanwhile, the extraction yield of dehydrator drying sample was optimized at one hr of extraction time with 10% yield 100% of ethanol concentration with 15.4% yield, 40:1 v/w of solvent ratio with 14.7% yield, and 48 hrs of drying with 8% yield. The optimum extract yield of dehydrator drying sample could be further applied for cosmeceutical application as it produced higher yield compared to oven drying sample.

1. Introduction

Watermelon [*Citrullus lanatus* (Thunb.)] is an important crop that belongs to the family Cucurbitaceae. Watermelon has recorded at fourth highest annual production of fruits in Malaysia with total production of 134,225 tonnes and planted area of 9,247 ha (Malaysia Department of Agriculture, 2020). Generally, the three main parts of watermelon are flesh, rind and seed. The composition of watermelon rind mainly consists of 13% (w/w) pectin, 10% (w/w) lignin, 23% (w/w) hemicellulose and 20% (w/w) cellulose depending on watermelon genotype (Ahamad *et al*., 2022).

Recent statistic shows that Malaysia throws away 17,000 tonnes of food waste per day (SWCorp Malaysia, 2022). Every food waste that get thrown away at landfill and incinerator may cause carbon dioxide emission and contributes towards pollution. Statistic by Municipal Solid Waste (MSW) Malaysia ascertained that 60% of food waste is contributed by the unused parts of fruits and vegetables, including watermelon rind during large quantities industrial processing in food and beverage industry. Watermelon rind is usually being discard and considered as waste due to its unappealing flavour. Watermelon waste can contribute and generate another new product which gives economic advantage such as high fibre watermelon rind flour (Adegunwa *et al*., 2019), biopolymer and food additive watermelon rind pectin source (Lee and Choo, 2020), bio sorbent material for wastewater (Lee and Choo, 2020; Ramakrishnan *et al*., 2020) and cosmeceutical anti-aging properties ingredient (Raikou *et al*., 2017).

Previous research utilizing the agro waste into

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eISSN: 2550-2166
Published by Rynnye Lyan Resources

valuable products such as cocoa pod husk and sugarcane bagasse in food packaging (Azmin *et al*., 2020), *Beta vulgaris* (Azmin *et al*., 2020) and overripen tomato into lip balm (Azmin, Abidin, Sulaiman *et al*., 2022), rice husk and rice straw into biochar (Selvarajh *et al*., 2020), banana peels into peel-off face mask (Azmin, Saidin, Nor *et al*., 2022), cocoa pod husk and kenaf into bioplastic (Azmin, Sharif, Nor *et al*., 2022) and many more. Therefore, watermelon waste has great potential to turn waste into resources by using the discarded watermelon rind as an alternative and low-cost ingredient in cosmeceutical products. The objective for this study comprise of determining optimized extraction conditions of watermelon rind using application of statistical design of experiment. The one-factor-at-a-time (OFAT) design evaluation were applied to observe the ethanol concentration, solvent-to-solid ratio, extraction time, and sample drying time variables for oven and dehydrator drying watermelon rind samples that providing the efficient extraction process and most optimized yield.

2. Materials and methods

2.1 Sample preparation

The watermelons [*Citrullus lanatus* (Thunb.)] with maturity index of 2 (70-90% ripped) and medium size classification (4-6 kg) were used. The red fleshy pulp and green hard skin were removed. The rind was cut into cube with 10 mm thickness and dried at 60°C in Memmert CTC256 hot air oven and BioChef Arizona Sol 6 tray food dehydrator. Five levels of sample drying time (24, 30, 36, 42 and 48 hrs) were conducted according to OFAT evaluation. The water content of dried watermelon rind samples were calculated using Equation 1. The samples were refined using Cosway Empress stainless steel grinder and Retsch sieve into 500 µm fine powder.

$$\text{Moisture content (\%)} = \left(\frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}}\right) \times 100 \quad (1)$$

2.2 Sonication extraction

The oven drying and dehydrator drying samples were extracted with ethanol in a flask placed in an ultrasonic bath sonicator (RS Pro, United Kingdom) with a frequency of 40 kHz and ultrasonic power 100 W. The top of the flask was covered by aluminium foil to minimize the evaporation of solvent. The samples were centrifuged at 2,800×*g* for 10 mins and filtered through 180 mm filter paper. The solvent was removed at 50°C via a rotary evaporation system. The extraction yield were calculated using Equation 2. All extracts were kept at -20°C prior to experimental.

2.3 One-factor-at-a-time evaluation of extract

$$\text{Extraction Yied (\%)} = \left(\frac{\text{Weight of obtained yield}}{\text{Weight of dry sample}}\right) \times 100 \quad (2)$$

One-factor-at-a-time (OFAT) design evaluation was applied to study one factor while the other variables were constant. Four experimental factors involved include ethanol concentration (20–100%), solvent-to-solid ratio (10:1-50:1 v/w), extraction time (0.5-3 hrs), and sample drying time (24-48 hrs). The factors range were chosen based on the previous method elaborated by Naknaen *et al*. (2016) and Yusof *et al*. (2020).

The fixed parameters were set at a solvent-to-solid ratio of 20:1 v/w, ethanol concentration of 80%, 3 hrs of extraction time and 48 hrs of sample drying time. The OFAT consist of ten independent runs for each variables as shown in Tables 1, 2, 3 and 4. OFAT was used to find out the most significant factors to be used before optimizing the variables using response surface methodology (RSM).

3. Results and discussion

One-factor-at-a-time (OFAT) was carried out to simplify the evaluation process by analysing a single factor to reduce the time and cost of the experiment. Figure 1 shows the influence of extraction time, ethanol concentration, solvent-to-solid ratio and sample drying time on the yield of watermelon rind after ten single

Table 1. The OFAT independent run for ethanol concentration factor.

Std	Block	Run	Ethanol concentration (%)
5	dehydrator drying	1	100
4	dehydrator drying	2	80
2	dehydrator drying	3	40
1	dehydrator drying	4	20
3	dehydrator drying	5	60
7	oven drying	6	40
9	oven drying	7	80
8	oven drying	8	60
6	oven drying	9	20
10	oven drying	10	100

Table 2. The OFAT independent run for solvent-to-solid ratio factor.

Std	Block	Run	Solvent-to-solid ratio (v/w)
1	dehydrator drying	1	10:1
5	dehydrator drying	2	50:1
2	dehydrator drying	3	20:1
3	dehydrator drying	4	30:1
4	dehydrator drying	5	40:1
9	oven drying	6	40:1
8	oven drying	7	30:1
6	oven drying	8	10:1
7	oven drying	9	20:1
10	oven drying	10	50:1

https://doi.org/10.26656/fr.2017.8(S2).61

Table 3. The OFAT independent run for extraction time factor.

Std	Block	Run	Extraction time (hrs)
1	Dehydrator drying	1	0.5
4	Dehydrator drying	2	2.0
3	Dehydrator drying	3	1.0
5	Dehydrator drying	4	3.0
2	Dehydrator drying	5	0.75
10	Oven drying	6	3.0
6	Oven drying	7	0.5
7	Oven drying	8	0.75
8	Oven drying	9	1.0
9	Oven drying	10	2.0

Table 4. The OFAT independent run for sample drying time factor

Std	Block	Run	Sample drying time (hrs)
5	Dehydrator drying	1	48
2	Dehydrator drying	2	30
3	Dehydrator drying	3	36
4	Dehydrator drying	4	42
1	Dehydrator drying	5	24
6	Oven drying	6	24
10	Oven drying	7	48
8	Oven drying	8	36
9	Oven drying	9	42
7	Oven drying	10	30

factor runs.

3.1 Effect of extraction time on watermelon rind yield

As illustrated in Figure 1(a), the extraction yield of watermelon rind increases as the sonication extraction time increases up to one hr of extraction time, and further increase of time reduces the yields ($p > 0.05$, R^2 = 0.7463, F value = 2.94). Oven drying and dehydrator drying sample at one hr of extraction time had achieved the maximum extraction yield with yield 15% and 10%, respectively. The result indicates that one hr is the maximum sonication extraction time as longer time resulted in the reduction of yields due to thermal degradation of the sample. This is because the ultrasonic process during extraction also acts to heat the fluid up to 42°C and the high sound and thermal energy might denature plant active constituents by producing free radicals (Abubakar and Haque, 2020).

Oreopoulou *et al.* (2019) recommended to performed sonication extraction using ethanol intervals from 15 to 45 min to preserve heat labile compounds like carnosic and rosmarinic acid. Yusuf *et al*. (2020) also extracted total phenolic compound from propolis using UAE process (20 kHz, 500 W) and found that 25 min sonication extraction time was the most optimal time for the sample. The optimized sonication extraction time were influences by the frequency and ultrasonic power used in the experiment.

3.2 Effect of ethanol concentration on watermelon rind yield

The extraction yield of the watermelon rind was highest at 100% ethanol concentration as presented in

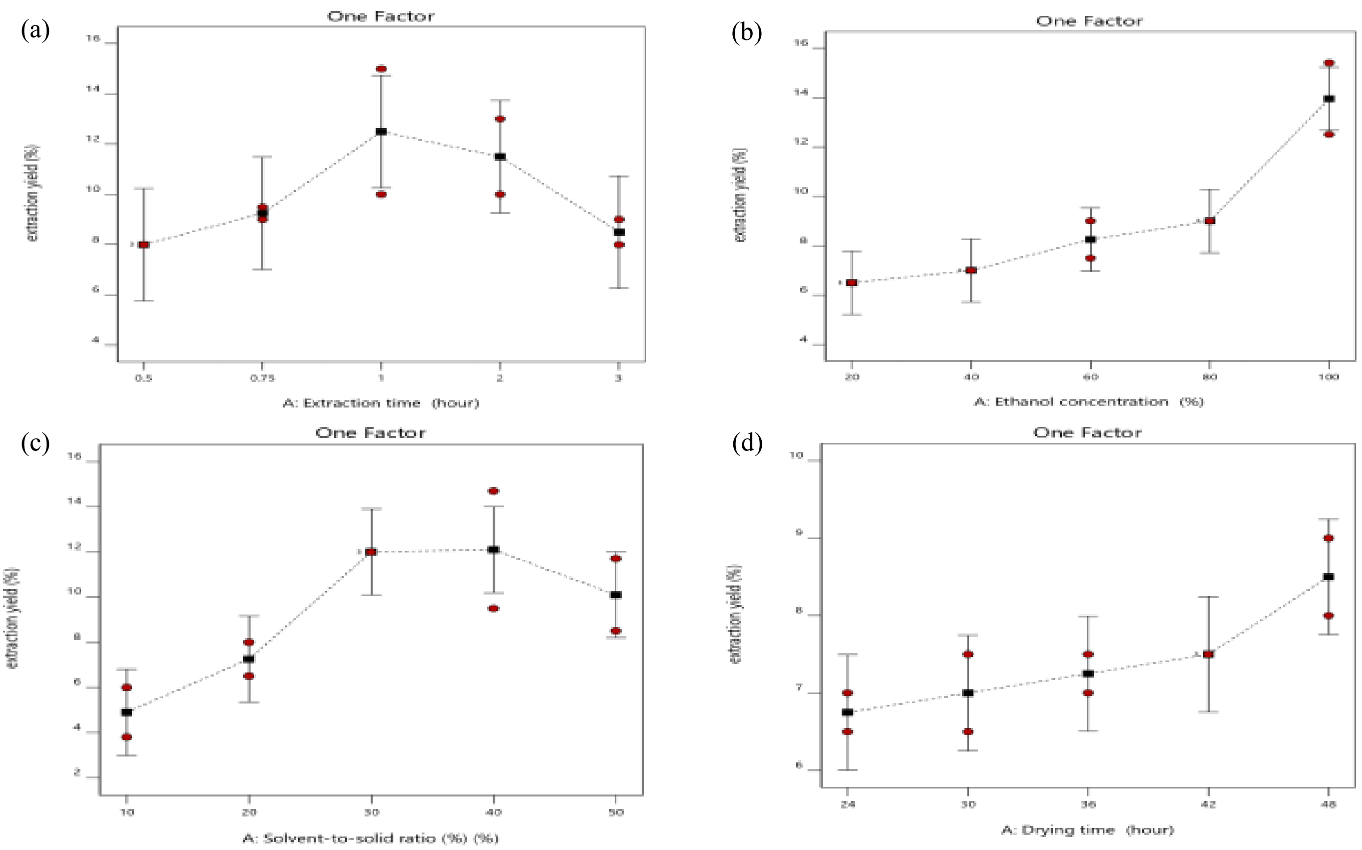

Figure 1. Extraction yield of watermelon rind at different parameter: (a) extraction time, (b) ethanol concentration, (c) solvent-to-solid ratio and (d) drying time.

https://doi.org/10.26656/fr.2017.8(S2).61

Figure 1(b). Ethanol concentration showed significant effects ($p < 0.05$, $R^2 = 0.9541$, F value = 20.80) on extract yield by escalating from 6.9% to 15.4% yield at 20% and 100% ethanol concentration, respectively. High ethanol concentration contributed to high recovery of extract. This result was consistent with Alara *et al*. (2018), which reported the extraction yield of *Vernonia amygdalina* leaf using MAE extraction process were increasing at 40% ethanol up to 80% ethanol concentration.

However, another point for consideration is that higher ethanol concentration would decreased in solvent polarity as the proportion of polar solvent decreased, thus decreasing the amount of polar antioxidant compound in extract (Azmin *et al*., 2016; Dzah *et al*., 2020). Ciğeroğlu *et al*. (2017) reported that higher polarity water mixture solvent is more effective in extracting TPC and TFP content of *Citrus unsiu* Marc. leaf extracts by UAE extraction process as 50% ethanol provide higher TPC yield than 100% ethanol. Similarly, Yusof *et al*. (2020) reported that the total phenolic content of propolis using UAE process were initially increased from 50% up to 80% ethanol, but reducing at 90% ethanol as the solvent polarity decreased. Contrary to Yusof *et al*. (2020), Kaderides *et al*. (2019) reported that 50% ethanol was more efficient than 70% ethanol in extracting phenolics from pomegranate peels by MAE process. This difference finding was associated by different extracting mechanism of irradiated power between UAE and MAE process.

To summarized, high ethanol concentration may not give positive effect as its leads to extraction of non-polar compound specifically some lipid (Yusof *et al*., 2020). Extracting polar antioxidant compound is a major concern in this study for final product development. Higher extraction yield did not lead to extraction efficiency if only extracting non-desirable compound (Azmin *et al*., 2015; Ahamad *et al*., 2022).

3.3 Effect of solvent-to-solid ratio on watermelon rind yield

Figure 1(c) exhibits the outcome of different solvent-to-solid ratio (1:10, 1:20, 1:30, 1:40 and 1:50) on watermelon rind extract yield. The extraction yield of watermelon rind was significantly increasing following the increase of solvent-to-solid ratio ($p<0.05$, $R^2 = 0.9455$, F value = 17.35) and reach the peak at 1:30 with a 12% extraction yield, then considerably decreased afterwards. Lower solvent-to-solid ratio results of too concentrated solvent to penetrate into plant cell wall. However, higher ratio also did not provide higher yield. Mohammadpour *et al*. (2019) opined that excessive solvent may cause in lowering the cavitation phenomena in ultrasonic extraction due to lower nucleation site between extracting solvent and plant sample.

Lee and Choo (2020) were optimized pectin extraction from watermelon rind and found that solvent-to-solid ratio did not have significant effect ($p>0.05$) on extraction yield. However, a study by Prakash Maran *et al*. (2014) reorted that microwave assisted extraction of pectin from watermelon rind were optimized at 1:20.3. The result was greatly influencing by the type of technique conducted to assisted the extraction process.

3.4 Effect of sample drying time on watermelon rind yield

Sample drying time did not show significant effect ($p>0.05$, $R^2 = 0.76$, F value = 3.17) to the watermelon rind extract using sonication extraction method. However, higher extraction yield (9%) can be observed at 48 hrs of drying time as shown in Figure 1(d). Higher temperature and longer drying time are put in consideration as watermelon rind has high amount of water content. Ho *et al*. (2018) reported that the antioxidant compounds in watermelon rind may denature if the sample does not properly dried as high-water content in sample may subjected to high level of enzymatic activity.

Petchsomrit *et al*. (2020) are using oven drying at 60°C until a stable weight obtained before conducting infusion technique resulting to only 1.422% watermelon rind lipid yield. Meanwhile, Lee and Choo (2020) used 60°C drying for 24 hrs for watermelon rind pectin extraction and were optimized at 8.38% yield. The result obtained by both studies were below than our optimized yield (9%).

4. Conclusion

Based on OFAT evaluation, it is obvious that the extraction time, ethanol concentration, solvent-to-solid ratio and sample drying time has strongly influenced on the yield of watermelon rind extract. The highest obtained yield for oven drying sample was at one hr extraction time, 100% ethanol concentration, 30:1 v/w ratio, and 48 hrs drying. Dehydrator drying sample was optimized at one hr of extraction time, 100% of ethanol concentration, 40:1 v/w of solvent ratio and 48 hrs of drying. The efficiency of watermelon rind sonication extraction was varied between different sample condition. Dehydrator drying sample shows significant higher ($p \leq 0.05$) extraction yield than oven drying sample. Thus, dehydrator sample were the best potential to be further used in cosmetic formulation.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgments

This study was financially supported by the Ministry of Education Malaysia for the Fundamental Research Grant Scheme (FRGS/1/2023/TK05/UMK/02/3) and Prototype Research Grant Scheme (PRGS/1/2023/TK02/UMK/02/1); and also Dana Inovasi Yayasan Muhibah Tan Sri Fng Ah Seng 2023 (R/TSF/A0700/01552A/005/2023/01181). These supports are gratefully acknowledged.

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