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Optimization of sweet potato pectin extraction using hydrochloric acid

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Abstract. Pectin has been used widely as thickener, stabilizer and gelling agent. However, the sources of pectin industrially are still limited. This study aims to optimize the extraction of pectin from sweet potato residue using hydrochloric acid. In this study, proximate analysis of the sweet potato residues were performed and the extraction of pectin from sweet potato residues using hydrochloric acid was optimized to maximize its yield using response surface methodology (RSM). Three parameters were manipulated and optimized which were temperature (°C), pH and extraction time (min). The extracted pectin was further analyzed for its degree of esterification (DE) using Fourier Transform Infrared Spectroscopy (FTIR). The sweet potato residue sample obtained moisture content of 79.7±1.7%, ash content of 1.08±0.09% and carbohydrate content of 34.3±2.7%. The optimum condition to extract pectin from sweet potato residues using hydrochloric acid was at extraction temperature 60 °C, pH 1 and extraction time 60 min with 23.48 % pectin yield. The pectin has 57.48% of DE which indicates high methoxyl pectin. The results show that hydrochloric acid can be used as one of the solvents to extract pectin from sweet potato.

1. Introduction

Pectin is natural hydrocolloids that are found in plants as main structural elements of cell walls. The source of commercial pectin are apple pomace and citrus peel, and to a certain extent sugar beet roots and sunflowers. Pectin is mainly used in food processing industry as gelling and thickening agents, emulsifiers as well as stabilizers in jams, jellies, confectionery products, and beverages. Other than that, pectin is also used in pharmaceutical and cosmetic industry as binding agent in the formulations and as carrier of a variety of drugs.

Extraction of pectin from a cheap and abundant renewable resource, such as agricultural and food waste is a concern, moreover it may improve waste management and create another prospect of income to the economy. This study aimed to convert sweet potato residue into added-value products for food application. Usually, this residue would be utilized for animal feed or discarded which would pollute the environment. Sweet potato, *Ipomoea batatas L.* (Lam.), contains high dietary fibers, minerals, vitamin and antioxidant such as carotene and phenolic acid. Sweet potato ranked as one of the most important crops in especially in developing countries besides rice, wheat, maize and cassava [1]. It has been reported that sweet potato residues contain starch, cellulose, hemicellulose, pectin, ash and other minor substances such as fat and protein [2].

Solvent extraction is the most common method to extract pectin. The yield and properties of pectin usually depends on the raw materials used to extract pectin and the extraction conditions, such as temperature, extraction time, pH, and type of extraction solvents [3]. Previous studies used acid [4, 5] or alkali [6, 7] for the extraction of pectin which has affected the percentage yield of pectin. In regards to extraction parameters, pH was considered as one of the most crucial parameters affecting the yield of pectin. Liu et al. has reported that pH significantly affected the yield of pectin extraction [8]. As the pH increase, the yield of pectin decreased [9-11]. Methacanon et al. has reported that extraction temperature was the most significant factor compared to other factors in their study of pectin extraction from *Pomelo albedo* (Citrus maxima) [10]. Adamu et al. has also reported that the higher the temperature, the higher the percentage yields of pectin [12]. Another significant factor that has been reported to affect pectin yield was extraction time [13, 14]. Pectin yield increase as extraction time increase because longer time provides more reaction opportunity [13].

Response surface methodology (RSM) was chosen as the tool to determine and investigate the interaction between factors for optimizing the extraction processes of pectin from the sweet potato residues. This study aimed to optimize the yield of pectin as the response by varying three factors namely temperature, pH and time using hydrochloric acid as the solvent. The pectin extracted from sweet potato residue at optimized condition was further analyzed to determine its degree of esterification.

2. Materials and methods

2.1. Materials and chemicals

The sweet potato (*Ipomoea batatas* var. Serdang 1) used in this work was obtained in Taman Universiti, Skudai, Johor. The sweet potato residues were washed and stored at 4˚C. Heat stable α-amylase from *Bacillus licheniformis* (Type XII-A, ≥500 units/mg protein, EC 3.2.1.1) and amyloglucosidase from *Aspergillus niger* (lyophilized powder, EC 3.2.1.3) were purchased from Sigma Aldrich. All chemicals used were of analytical grades.

2.2. Sweet potato residue pre-treatment

The residue was washed under tap water to remove dirt. The residue was cut into small pieces before being ground. Ground sweet potato residue was dried in the oven at 50°C for 12 h.

2.3. Sweet potato residue analysis

Analysis was conducted to determine the physicochemical properties of the sweet potato residue. The residue was analyzed for moisture content, ash and carbohydrate contents.

2.3.1. Moisture content analysis. The sweet potato residue was cut into smaller pieces and placed in the moisture analyzer MX-50 (AND Company, Japan). The sample was added until the weight was two grams. The moisture analyzer was set to 180ºC. After 20 min, the moisture content was recorded. The procedure was repeated for three times.

2.3.2. Carbohydrate Content. The carbohydrate content of sweet potato residues was determined using phenol sulphuric acid method [15]. First, the standard curve of carbohydrate content was generated using glucose standard solution (100 mg glucose/L). Standard stock solution with concentration of 0.1 mg/L was made for standard curve. The 5% phenol solution was prepared by mixing 5 mL of phenol with 95 mL of distilled water. Ten test tubes with each having 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9 and 1.0 mL of stock solution were prepared respectively. Final volume of 1 mL was prepared by adding distilled water. The distilled water was used as reagent blank. Phenol (1 mL) and 98% sulphuric acid (5 mL) was added into each tube. The absorbance was obtained at 490 nm. The steps were repeated using sample of sweet potato residues.

2.3.3. Ash analysis. Two grams of dried sweet potato residue was weighed. Then, it was put into porcelain bowl and placed into furnace at 550 °C for 6 h. After that, the sample was weighed again.

2.4. Preparation of cell wall material

Cell wall material (CWM) was prepared from sweet potato residue according to the previous published method [4]. Ground dried sweet potato residue (10 g) was put into distilled water (200 mL) and boiled for 5 min. The suspension was maintained at 80°C, and 0.5 mL of heat-stable α-amylase was added, and then incubated for 30 min to hydrolyze the residual starch. The mixture was centrifuged at 3000 rpm for 10 min, and then the supernatant was discarded. The digestion of the residue was repeated with the addition of 0.5 mL amyloglucosidase. The mixture was filtered using two layers of cheesecloth. The residue was washed with distilled water, methanol and acetone allowed to air-dried

2.5. Extraction of pectin

Pectin was extracted from the flask containing 10 g of dried CWM, added with 50 mL pH 1 hydrochloric acid (HCl). The flask was stirred for the sample and HCl to mix properly at 60°C for 30 min. The resulting slurries was allowed to cool to room temperature and then centrifuged at 5000 rpm for 30 min to remove solid particles. The supernatant that contain pectin was filtered through double layer cheesecloth. The concentrated filtrates were precipitated with 95% ethanol and kept at 4°C. The coagulated pectin was centrifuged at 5000 rpm for 30 min. The precipitate of pectin was washed with 60%, 75% and 90% ethanol and centrifuged at 5000 rpm for 10 min to remove the other saccharides. The pectin obtained was dispersed in distilled water with gentle stirring and then subjected to freeze drying. The pectin extraction steps were repeated under different conditions (according to the experimental design in Table 1). The yield of pectin percentage was determined as gram of product obtained per 10 g of dried cell wall material used as shown in equation (1).

$$\text{Yield of pectin (\%)} = \frac{\text{Weight of dried extracted pectin (g)}}{\text{Weight of dried cell wall material (g)}} \times 100 \tag{1}$$

2.6. Experimental design of extraction of pectin from sweet potato residue

In this study, a Box-Behnken Design (BBD) with three independent variables at three levels and five replications of the centre points was employed to optimize and explore the effect of extraction conditions (A: extraction temperature; B: pH of acid solution; C: extraction time) on the response i.e. yield (%) of pectin from sweet potato residue. The variables chosen were known to have significant on the yield of pectin. The experimental design (Table 1) was generated using Design Expert software Version 6.0 and has 17 experimental runs that included five centre points to assess the pure error. The data obtained were fitted using an empirical second order polynomial equation as shown in equation (2) in order to express the relationship between the independent variables and response [16].

$$Y_i = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} {X_i}^2 + \sum_{i,j}^{k} \beta_{ii} X_i X_j \tag{2}$$

Y_i is the predicted response, X_i and X_j are the independent variables, k is the number of independent variables, β_o is the constant, β_i is the linear coefficient, β_{ii} is the quadratic coefficient and β_{ij} is the interaction coefficient. The model was verified by performing analyzes of variance (ANOVA) to test the significance of the model, testing of lack of fit and prediction error sum of squares (PRESS) residuals to determine the coefficient of determination (R^2).

2.7. Analysis of pectin

2.7.1. Degree of Esterification of Pectin. The degree of esterification (DE) of pectin from sweet potato residue was analyzed using Fourier Transform Infrared Spectroscopy FTIR (Perkin Elmer, USA) to determine the main functional groups [17]. Degree of esterification was calculated as the number of esterified carboxylic groups / number of total carboxylic groups as shown in equation (3).

$$DE\% = \frac{\text{Absorbance at peak around } 1740\ cm^{-1} \times 100\%}{\text{Absorbance of peak around } 1740\ cm^{-1} + 1639\text{-}1600\ cm^{-1}} \quad (3)$$

Two specific frequencies, namely the bands at 1740 and 1630 cm^{-1} were used in order to calculate the DE. The carboxyl ester groups 32 absorb at about 1740 cm^{-1} whereas the corresponding carboxylate groups absorb at about 1600 cm^{-1}. The ratio of the areas of the bands at 1740 cm^{-1} over the sum of the areas of the bands at 1740 and 1630 cm^{-1} should be proportional to the DE.

2.7.2. Statistical Analysis. The results from antioxidant activity of pectin were analyzed using t-test by using data analysis tool in Microsoft Excel. The value of $P<0.05$ indicates significant differences between means of group studied. The standard deviations of the measurements of duplicates were presented by the error bars in the chart.

3. Results and discussion

3.1. Analysis of sweet potato residue

The moisture content of the sweet potato residue sample was 79.7±1.7% which shows high amount of moisture. The value obtained agrees with the data reported previously [18, 19]. Ginting and Yulifianti also stated that sweet potato tends to have high moisture content [20]. The ash content of sample was 1.08±0.09% similar to the value reported by Adepoju and Adejumo [21]. The low ash content would means that the sweet potato residue may lack in some minerals [18]. The carbohydrate content of sample was 34.3±2.7%. Previous study by Adepoju and Adejumo has reported almost similar value of carbohydrate content [21].

3.2. Optimization of pectin yield extraction using hydrochloric acid

3.2.1. Model Fitting. The response for pectin yield ranged from 3% to 24.4% of sweet potato residue as shown in Table 1. A response surface analysis and analysis of variance (ANOVA) were employed to determine the regression coefficients, statistical significance of the model terms and to fit the mathematical models of the experimental data that aimed to optimize the overall region for response variable.

Table 1. Experimental design with three factors and three levels and the pectin yield response under different extraction condition.

Run	(A) Temperature	(B) pH	(C) Time (min)	Pectin Yield (%)
1	75	1	90	19.7
2	60	2	30	6.1
3	60	3	60	9.8
4	75	3	30	5.0
5	75	3	90	9.0
6	60	1	60	24.4
7	75	2	60	6.7
8	90	2	30	6.15
9	75	2	60	6.8
10	90	1	60	16.2
11	75	2	60	6.9
12	90	3	60	9.75
13	75	2	60	3.0
14	60	2	90	8.9
15	75	1	30	13.6
16	75	2	60	6.3
17	90	2	90	15.3

The following regression equation (4) was the empirical model of the studied pectin yield in terms of coded factor. The model was reduced to a simplified regression equation to eliminate the insignificant factor interactions. Table 4 shows ANOVA result for the model.

$$\text{Yield} = 5.93 - 0.22A - 5.04B + 2.76C + 3.19A^2 + 5.90B^2 + 2.04AB + 1.59AC \quad (4)$$

It was observed that regression was significant as $P<0.05$. Therefore, the reduced quadratic model can be used as response surface for extraction of pectin. The quality of fit to the second-order polynomial models for yield of pectin was established based on the coefficients of determination (R^2), which was 0.9248, adjusted R^2 of 0.8663 and predicted R^2 of 0.4441. The ‘‘fitness’’ of the model was studied through the lack-of-fit test ($P>0.05$), which indicated the suitability of models to accurately predict the variation as shown in Table 2. Fig. 1 shows the predicted versus actual values and normal plot for residuals which indicate the fitness of the model for the prediction of the extraction of pectin yield. As expressed in equation (3), extraction time (C) showed significantly linear effects ($P<0.05$) on the pectin yield, which was clearly indicated by the large positive linear regression coefficient (+2.76) while pH (B) possessed a significantly negative effect ($P<0.05$) with the linear coefficient of -5.04. Extraction temperature (A) has a negative linear coefficient (-0.22) but not significant ($P>0.05$).

Table 2. ANOVA table for the regression model of pectin yield.

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	Remarks
Model	491.01	7	70.14	15.81	0.0002	*significant*
A	0.41	1	0.41	0.091	0.7694	
B	203.52	1	203.52	45.86	< 0.0001	
C	60.78	1	60.78	13.70	0.0049	
A^2	43.00	1	43.00	9.69	0.0125	
B^2	147.17	1	147.17	33.17	0.0003	
AB	16.61	1	16.61	3.74	0.0851	
AC	10.08	1	10.08	2.27	0.1660	
Residual	39.94	9	4.44			
Lack of Fit	28.93	5	5.79	2.10	0.2458	*not significant*
Pure Error	11.01	4	2.75			
Cor Total	530.95	16				

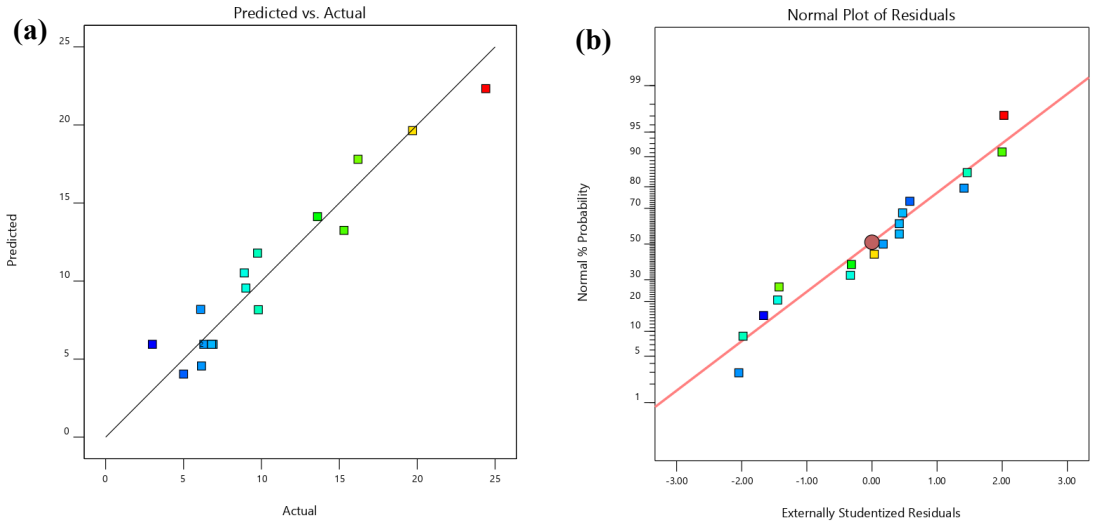

Figure 1. Graph of (a) predicted versus actual values of response (pectin yield) and (b) normal plot of residuals

3.2.2. Analysis of the Response Surface Plots. Fig. 2 illustrates the three dimensional (3D) plots by presenting the response as a function of two factors and keeping the other constant at its centre level. The results show that the increase of pH causes a decrease in pectin yield. The effects of interaction between extraction temperature and pH at different extraction time on the pectin yield are shown in Fig. 2 (a-c). The results show that yield increase as the pH decrease at all temperature range from 60 to 90°C. This result is in agreement with the result reported by Hamidon & Abang Zaidel where higher pectin was extracted from sweet potato residues at pH lower than 2 [5]. This may be due to the effect at high temperature and low pH that might prompt the disruption of hydrogen bonds and ester linkages between pectin and cell wall and then increase the rate of diffusion and pectin extraction [22-24]. According to BeMiller, the increase of hydrogen ion concentrations, repressed the ionization of carboxylate groups, in corresponding to lower pHs [25]. Thus, the highly hydrated carboxylate group are converted into slightly hydrated carboxylate acid groups. As a result of losing some charges cause

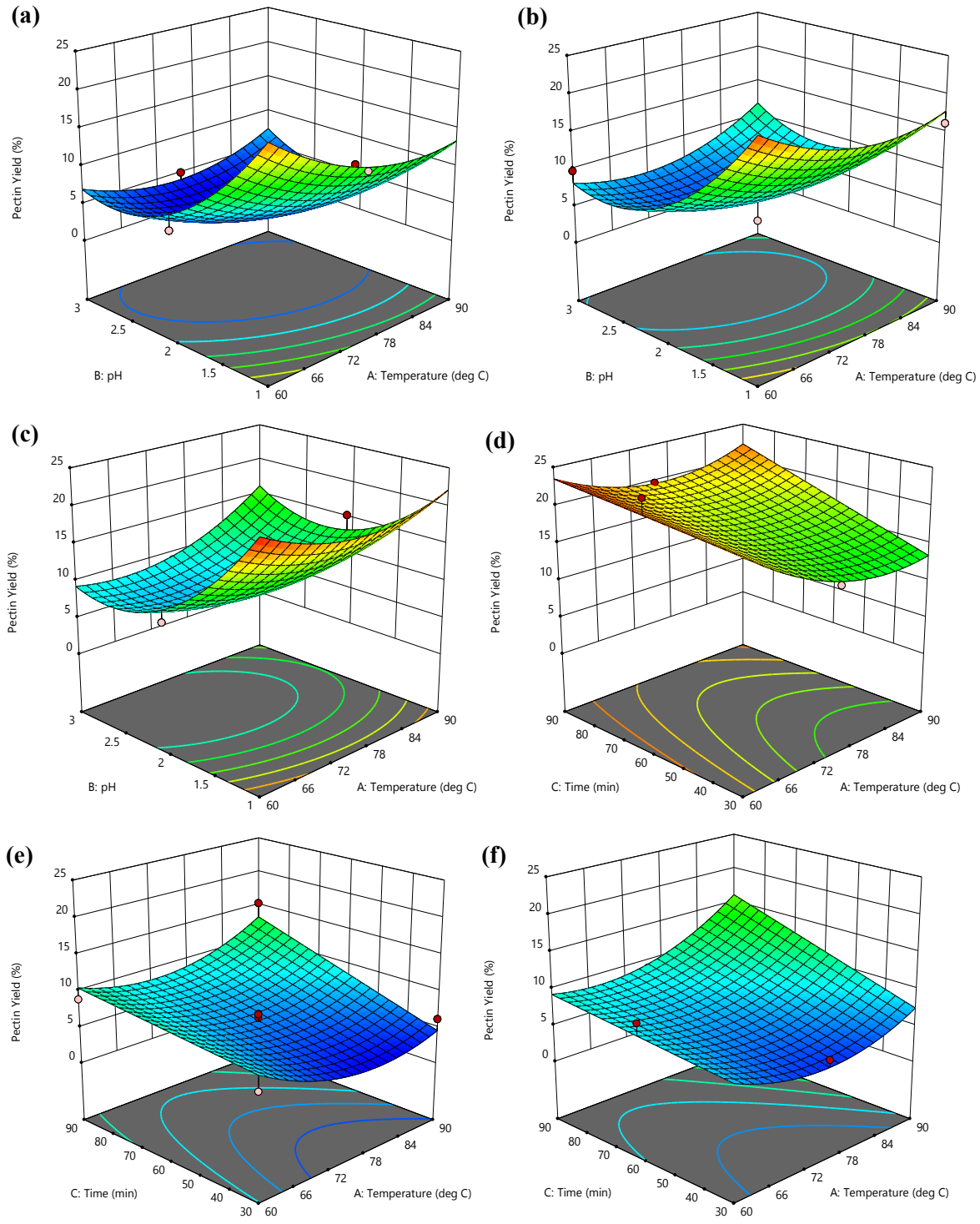

Figure 2. Response surface plots for the effect of temperature (A) and pH (B) at extraction time of (a) 30 min (b) 60 min (c) 90 min and the effect of temperature (A) and extraction time (C) at (d) pH 1 (e) pH 2 (f) pH 3

the polysaccharide molecules to become less repulsive, which can help pectin to form gel, giving more precipitated pectin at lower pH [8].

Fig. 2 (d-f) shows the effect of interaction between extraction temperature and extraction time on the pectin yield at different pH. The result shows that the yield increases as the extraction time increase but the yield decrease when extraction temperature increases. It has also been reported that the extended time favours the recovery of pectin [24]. This might due to the time requirement for the full release of pectin within an acidic medium where the liquid has to penetrate into the cell wall material. It was expected that at higher temperature will produce a high yield of pectin. However, in this study the optimum temperature to obtain highest yield of pectin was at 60°C. This is not in agreement with previous reported study by Abang Zaidel et al. where the maximum recovery of pectin yield was obtained at 90°C using hydrochloric acid [7]. This might be due to the interaction with other factors such as pH and time.

From the model, optimum yield of pectin obtained was 23.48%. In comparison with alkaline method, acid extraction method produce higher yield of pectin. In previous study, Abang Zaidel et al. has reported the highest percentage yield of pectin extracted using alkaline method at 16.78% using 0.25 M NaOH [7], while Nurdjanah reported that 0.05 M NaOH yielded 11.1% of pectin [6].

3.2.3. Model Validation. The model was verified based on the result obtained from response surface analysis, the optimal extraction conditions from the model were at temperature of 60°C, extraction time of 89.36 min and pH 1 solution that give pectin yield of 23.48%. The validation was conducted under the conditions of 60°C, extraction time of 90 min and pH 1 solution that produce pectin yield of 24.41%, which was in reasonable agreement with the predicted value ($P<0.05$). Therefore, the model was adequate and accurate for this study.

4. Conclusion

In conclusion, this study found that the sweet potato residue sample obtained moisture content of 79.7±1.7%, ash content of 1.08±0.09% and carbohydrate content of 34.3±2.7%. The optimum extraction condition to extract pectin from sweet potato residues using hydrochloric acid was at temperature of 60°C, extraction time of 90 min and pH 1 solution that give pectin yield of 23.48%. The pectin has 57.48% of DE which indicates high methoxyl pectin. This study also shows that acid extraction using hydrochloric acid has high potential to be used in the pectin extraction from sweet potato residues.

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