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Microwave-assisted extraction of pectin from grape pomace

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The utilization of microwave technique for the pectin extraction from grape pomace (Fetească Neagră and Rară Neagră), its influence on yield, galacturonic acid content, degree of esterification and molecular weight of pectin were analyzed. The optimal conditions of the extraction process were microwave power of 560 W, pH of 1.8 for 120 s. The pectin samples extracted by MAE in optimal conditions were analyzed by comparing with commercial apple and citrus pectin based on FT-IR analysis, thermal behavior, rheological characteristics and microstructure. The FT-IR analysis established the presence of different functional groups which are attributed to the finger print region of extracted pectin, while the rheological behavior presented a good viscoelasticity of pectin solutions. The obtained data assumes that grape pomace has a great potential to be a valuable source of pectin which can be extracted by simple and quick techniques, while maintaining analogous quality to conventional sources of pectin.

Pectin is a family of natural polysaccharides of α-*D*-(1,4) galacturonic acid (GalA) present which are found in the plant cell walls (CWs) mostly in fruits, such as citrus fruits and apples, etc. and vegetables[1,2]. Besides, the main sources of pectin, other agricultural food by-products such as cocoa pod husks[3,4], tomato waste[5], potato pulp[6], watermelon rind waste[7,8], persimmon peel[9], grape pomace[10,11] were used for extraction, and the obtained pectin yield and its structural characteristics were investigated and stated. According to the regulation of Food and Agriculture Organization of the United Nations (FAO), pectin must contain more than 65% GalA, to be considered as being „desirable" in terms of quality[12].

Usually, the production of pectin includes extraction, purification and drying. One of the most significant factors which influence the yield and quality of pectin is the use of a suitable method of extraction[13]. In the industry, the most commonly technique for the pectin extraction is conventional method which implicates the use of hot water (60–100 °C) acidified to a pH 1.5–3.0[14,15], but different alternative methods have been implemented to maximize the pectin yield, such as enzyme-assisted[16,17], microwave-assisted[18,19], subcritical fluid[20,21] and ultrasound pulses[17,19].

The microwave-assisted extraction (MAE) is a technique which combines the use of microwave energy with that of a heat solvent to partition analytes from various sample matrices[22], presenting a lot of advantages, such as high efficiency, low energy consumption, short processing time, low cost, cleanliness, easy controllability and low solvent requirements[19]. MAE enhances the water absorption capacity and capillary-porous components of the plant CW[23,24]; these modification contribute an opportunity to increasing the extraction yield of different analytes from plant CW, such as cellulose, hemicellulose and pectin[23]. Moreover, the energy causes the vibration of polar molecules with accelerated enhance of temperature which increases the extraction yield[25]. MAE has shown more efficient for the pectin extraction compared with conventional methods[14,25].

In this study, pectin was extracted from grape pomace of two varieties, Fetească Neagră and Rară Neagră (*Vitis vinifera* L.), by applying microwave technique. This is the first research about utilization of a non-conventional extraction method, i.e. microwave-assisted extraction, to extract pectin from this by-product. The obtained pectin was described in terms of yield, galacturonic acid content, molecular weight and degree of esterification. Therefore, the aim of this study was to optimize the extraction process, to comprehend the effect of the working parameters on the yield and physicochemical characteristics of grape pomace pectin and correlated with those of commercial apple and citrus pectin.

Materials and methods

Materials. Grape pomace was collected by processing two different *Vitis vinifera* L. varieties (Fetească Neagră and Rară Neagră) from 2019 harvest, cultivated in the Bugeac area, Republic of Moldova. The grape pomace was dried in an oven at 50 °C until constant weight, then it was powdered and sieved in order to obtain 125–200 μm of particle size.

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Methods.   All methods were performed in accordance with the relevant guidelines and regulations.

Extraction of pectin from dried grape pomace using MAE.   MAE was performed in a household microwave oven Gorenje MO 17 DW (Gorenje, Velenje, Slovenia) at working frequency of 2450 MHz with regulable time and microwave power. 10 g of dried grape pomace was placed into bottle and ultrapure (Milli-Q) water containing different pH values (1, 2 and 3) at s solid–liquid ratio of 1:10 (w/v). The pH of the solvent was adjusted with citric acid. The bottle was fixed in the middle of the oven over a rotating dish and was subjected to microwave radiation at different powers (280, 420 and 560 W) for the selected exposure time (60, 90 and 120 s). After extraction procedure, the mixtures were allowed to cool down to room temperature (25 °C) and separated by centrifugation (35 min at 3500 rpm), followed by the precipitation procedure with an equal volume of 96% (v/v) ethanol. Finally, pectin was purified with 96% (v/v) ethanol for three times and dried to a constant weight at 50 °C in an oven.

Pectin yield.   Pectin yield was determined using Eq. (1):

$$Yield\ (\%) = \frac{m_0}{m} \times 100 \tag{1}$$

where m_0, weight of dried pectin (g); m, weight of dried grape pomace powder (g)[18].

Galacturonic acid content.   The galacturonic acid (GalA) content of samples was measured using the sulfamate/m-hydroxydiphenyl method developed by Filisetti-Cozzi[26] and Melton & Smith[27]. Sample preparation was made according to Miceli-Garcia[28] and Dranca & Oroian[29]. The absorbance for each sample was read at 525 nm against the reageant control with a UV-3600 Plus UV–Vis–NIR spectrophotometer (Shimadzu Corporation, Kyoto, Japan).

Degree of esterification.   The degree of esterification (DE) of samples was determined by the titrimetric method described by Franchi[30] and Wai et. al.[31]. The DE was calculated with the Eq. (2):

$$DE\ (\%) = \frac{V_2}{V_1 + V_2} \times 100 \tag{2}$$

where V_1, volume of sodium hydroxide used for the first titration (mL); V_2, volume of sodium hydroxide used for the second titration (mL).
    The DE of pectin samples was measured in triplicate.

Molecular weight.   Molecular weight (M_w) of samples was carried out by high-performance size-exclusion chromatography using a HPLC system (Shimadzu Corporation, Kyoto, Japan) equipped with a LC-20 AD liquid chromatograph, SIL-20A auto sampler, a Yarra 3 μm SEC-2000 column (300 × 7.8 mm; Phenomenex, Torrance, CA, USA) and coupled with a RID-10A refractive index detector (Shimadzu Corporation, Kyoto, Japan). The samples were made according to Dranca et al.[18].

Color.   The color of the pectin samples was analyzed in triplicate at 25 °C with a CR-400 chromameter (Konica Minolta, Tokyo, Japan). CIE L*, hue (h^*_{ab}) and chroma (C^*_{ab}) were obtained from the reflection spectra of the pectin samples with illuminant D65 and 2° observer.

FT-IR analysis.   The samples extracted by MAE in optimal conditions (FN and RN pectin) and the commercial samples (apple and citrus pectin) were conducted to to FT-IR analysis using a Spectrum Two infrared spectrophotometer (PerkinElmer, Waltham, MA, USA). The distinctive spectra were registered (three scans for each sample) in the frequency range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1}[32].

Thermal analysis.   Thermal analysis of pectin (samples extracted by MAE in optimal conditions, commercial apple and citrus pectin) was lead to differential scanning calorimetry technique (DSC); the measurement was repeated three times. DSC analysis of 1 mg dried pectin weighted in aluminum pan and placed in the apparatus (DSC 25, TA Instruments, New Castle, DE, USA) with an empty pan used as reference, were accomplished over a temperature range of 0–300 °C at a heating rate of 10 °C/min with a flow rate of 50 mL/min.

Rheological characterization of pectin solutions.   In order to obtain 5% (w/w) solutions, pectin sample extracted by MAE in optimal conditions, commercial apple and citrus pectin were homogenized using Milli-Q water adjusted to pH 4 under constant stirring at 40 °C for 12 h. Then, the samples were cooled to room temperature (25 °C) and stocked under refrigeration at 4 °C for 16 h.
    The dynamic viscosity of pectin solutions was determined with a Haake Mars 40 rheometer (Thermo Fisher Scientific, Waltham, MA, USA) using a cone (Ø 35 mm, 2°)—plate system at 20 °C. During measuring the dynamic viscosity (η, Pa·s) and shear stress (τ, Pa), the shear rate ($\dot{\gamma}$, s^{-1}) was ranged between 0 and 100 s^{-1}. The stress sweeps of loss modulus (G′, Pa) and elastic modulus (G″, Pa) were determined at 1 Hz for measurement of the viscoelastic region. The frequency was presented a range from 0.1 to 100 Hz and the stress was selected within the linear viscoelastic region.

Variables	Levels		
	−1	0	1
Microwave power (W)	280	420	560
Irradiation time (s)	60	90	120
pH	1	2	3

Table 1. Variables and levels used for Box-Behnken design.

The 'creep and recovery' analysis was measured at a constant stress of 1 Pa, which was implemented and maintained for 180 s; the stress was released to accept sample recovery for 180 s. The creep parameters were determined using Eq. (3):

$$J(t) = \frac{\gamma(t)}{\sigma} \tag{3}$$

where J, creep compliance; σ, constant stress over time (t); γ, shear deformation.

Microstructure. The structural morphology of the pectin samples was analyzed by scanning electron microscopy (SEM; SU-70, Hitachi, Tokyo, Japan). Powder of pectin was placed on the sample table with conductive double-sided adhesive carbon tape and analyzed using an accelerating voltage of 30 kV at different magnifications (150×, 400× and 700×).

Statistical analysis. In this sudy, a three-factor full factorial Box-Behnken design was adjusted in order to analyse and optimize the influence of the independent variables, power (X_1), pH (X_2) and time (X_3) on the extraction yield, DE, GalA content and M_w of pectin. The coded levels of the variables are presented in Table 1. All graphics and calculations were accomplished utilizing the statistical software Design Expert 13 (trial version, Minneapolis, MN, USA); the analysis was rehearsed in triplicate. The ANOVA test was used to evaluate the difference between means at the 95% confidence level ($p < 0.05$) with Fisher's least significant difference procedure.

Results and discussion

Model fitting and statistical analysis.
The microwave-assisted extraction (MAE) of pectin from Fetească Neagră (FN) and Rară Neagră (RN) grape pomace was modeled utilizing the Box-Behnken design with three parameters in accord with the data presented in Table 2. Each independent variable had three levels, as follows: microwave power (280, 420 and 560 W), irradiation time (60, 90 and 120 s) and pH (1, 2 and 3). The responses of the design were extraction yield (Y, %), galacturonic acid content (GalA, g/100 g), degree of esterification (DE, %) and molecular weight (M_w, g/mol) of pectin.

The model applied to predict the evolution of the responses was a quadratic (second order) polynomial response surface model which was used to fit the results accomplished by design; the data of the analysis of variance (ANOVA) was presented in Table 3. The square polynomial equations that characterized the combined influence of microwave power (X_1), irradiation time (X_2) and pH (X_3) on extraction yield, GalA, DE and M_w are presented below.

$$Y_{FN}(\%) = 6.79 + 0.97 \cdot X_1 + 0.64 \cdot X_2 - 1.24 \cdot X_3 + 0.54 \cdot X_1 \cdot X_2 - 0.42 \cdot X_1 \cdot X_3$$
$$- 0.59 \cdot X_2 \cdot X_3 - 0.65 \cdot X_1^2 + 0.30 \cdot X_2^2 - 0.69 \cdot X_3^2 \tag{4}$$

$$GalA_{FN}(\text{g/100 g}) = 66.3 + 6.56 \cdot X_1 + 4.7 \cdot X_2 - 7.87 \cdot X_3 + 3.87 \cdot X_1 \cdot X_2$$
$$- 4.52 \cdot X_1 \cdot X_3 - 3.47 \cdot X_2 \cdot X_3 - 4.67 \cdot X_1^2 + 1.18 \cdot X_2^2 - 3.68 \cdot X_3^2 \tag{5}$$

$$DE_{FN}(\%) = 72.9 + 3.95 \cdot X_1 + 3.22 \cdot X_2 - 4.82 \cdot X_3 + 2.36 \cdot X_1 \cdot X_2$$
$$- 2.99 \cdot X_1 \cdot X_3 - 1.94 \cdot X_2 \cdot X_3 - 3.11 \cdot X_1^2 + 1.43 \cdot X_2^2 - 2.11 \cdot X_3^2 \tag{6}$$

$$M_{w\,FN}(\text{g/mol}) = 44{,}635 + 1091.25 \cdot X_1 + 419 \cdot X_2 - 1302 \cdot X_3 + 96.75 \cdot X_1 \cdot X_2$$
$$- 803.25 \cdot X_1 \cdot X_3 - 369.75 \cdot X_2 \cdot X_3 - 827.88 \cdot X_1^2 - 60.38 \cdot X_2^2 - 1050.87 \cdot X_3^2 \tag{7}$$

$$Y_{RN}(\%) = 8.42 + 1.09 \cdot X_1 + 0.72 \cdot X_2 - 1.37 \cdot X_3 + 0.34 \cdot X_1 \cdot X_2 - 0.23 \cdot X_1 \cdot X_3$$
$$- 0.72 \cdot X_2 \cdot X_3 - 0.8 \cdot X_1^2 + 0.49 \cdot X_2^2 - 0.93 \cdot X_3^2 \tag{8}$$

$$GalA_{RN}(\text{g/100 g}) = 67.85 + 7.48 \cdot X_1 + 5.21 \cdot X_2 - 7.72 \cdot X_3 + 4.31 \cdot X_1 \cdot X_2$$
$$- 5.46 \cdot X_1 \cdot X_3 - 3.29 \cdot X_2 \cdot X_3 - 4.90 \cdot X_1^2 + 1.78 \cdot X_2^2 - 4.16 \cdot X_3^2 \tag{9}$$

	Independent variables			Measured response				Predicted response			
Run	Microwave power (W)	Irradiation time (s)	pH	Y (%)	GalA (g/100 g)	DE (%)	M_w (g/mol)	Y (%)	GalA (g/100 g)	DE (%)	M_w (g/mol)
FN											
1	280	90	3	3.96	50.92	62.28	4.15×10^4	3.66	48.04	61.89	4.11×10^4
2	560	90	3	4.85	51.87	63.01	4.19×10^4	4.77	52.12	63.82	4.17×10^4
3	560	120	2	9.03	81.24	82.29	4.57×10^4	8.61	77.93	80.74	4.53×10^4
4	280	90	1	5.21	54.98	66.38	4.20×10^4	5.29	54.73	65.57	4.21×10^4
5	420	90	2	6.47	62.48	70.94	4.43×10^4	6.79	66.30	72.90	4.46×10^4
6	280	120	2	5.79	57.24	64.48	4.31×10^4	5.58	57.07	68.13	4.29×10^4
7	420	90	2	6.26	64.84	72.07	4.41×10^4	6.79	66.30	72.90	4.46×10^4
8	420	90	2	7.32	70.36	76.18	4.55×10^4	6.79	66.30	72.90	4.46×10^4
9	280	60	2	4.94	52.11	64.87	4.19×10^4	5.36	55.42	66.42	4.23×10^4
10	420	90	2	6.89	65.28	72.16	4.42×10^4	6.79	66.30	72.90	4.46×10^4
11	560	60	2	6.02	60.63	69.24	4.41×10^4	6.23	60.80	69.59	4.43×10^4
12	560	90	1	7.78	74.02	79.05	4.56×10^4	8.08	76.91	79.44	4.59×10^4
13	420	60	1	6.91	66.56	72.63	4.45×10^4	6.40	63.50	71.89	4.40×10^4
14	420	90	2	7.02	68.53	73.14	4.49×10^4	6.79	66.30	72.90	4.46×10^4
15	420	120	1	8.75	79.42	81.05	4.56×10^4	8.88	79.84	82.21	4.56×10^4
16	420	60	3	5.24	55.31	67.29	4.21×10^4	5.11	54.71	66.13	4.21×10^4
17	420	120	3	4.72	54.09	67.93	4.17×10^4	5.23	57.15	68.67	4.22×10^4
RN											
1	280	90	3	4.81	51.09	62.14	4.25×10^4	4.47	49.04	60.99	4.21×10^4
2	560	90	3	5.78	53.27	63.02	4.26×10^4	6.16	53.09	63.21	4.24×10^4
3	560	120	2	11.23	85.18	83.11	4.60×10^4	10.26	81.71	81.45	4.56×10^4
4	280	90	1	7.11	53.39	65.68	4.28×10^4	6.72	53.57	65.48	4.31×10^4
5	420	90	2	8.21	63.93	71.74	4.49×10^4	8.42	67.85	72.94	4.51×10^4
6	280	120	2	7.65	59.74	69.49	4.39×10^4	7.41	58.13	69.18	4.37×10^4
7	420	90	2	8.32	66.65	72.17	4.47×10^4	8.42	67.85	72.94	4.51×10^4
8	420	90	2	8.78	71.22	75.83	4.59×10^4	8.42	67.85	72.94	4.51×10^4
9	280	60	2	5.67	52.88	63.99	4.28×10^4	6.64	56.35	65.64	4.31×10^4
10	420	90	2	8.41	67.35	71.33	4.48×10^4	8.42	67.85	72.94	4.51×10^4
11	560	60	2	7.89	61.07	70.02	4.44×10^4	8.13	62.67	70.32	4.46×10^4
12	560	90	1	9.03	77.39	79.08	4.58×10^4	9.37	79.44	80.22	4.62×10^4
13	420	60	1	8.49	68.34	72.19	4.48×10^4	7.90	64.69	70.73	4.42×10^4
14	420	90	2	8.37	70.08	73.65	4.50×10^4	8.42	67.85	72.94	4.51×10^4
15	420	120	1	10.16	80.26	82.26	4.59×10^4	10.79	81.68	82.76	4.59×10^4
16	420	60	3	7.24	57.25	65.19	4.26×10^4	6.61	55.83	64.68	4.27×10^4
17	420	120	3	6.03	56.01	65.87	4.21×10^4	6.62	59.66	67.32	4.27×10^4

Table 2. Box–Behnken experimental design matrix with measured and predicted values. *FN* Fetească Neagră, *RN* Rară Neagră, *Y* yield, *GalA* galacturonic acid content, *DE* degree of esterification, *M_w* molecular weight.

$$DE_{RN}(\%) = 72.94 + 4.24 \cdot X_1 + 3.67 \cdot X_2 - 5.37 \cdot X_3 + 1.9 \cdot X_1 \cdot X_2$$
$$- 3.13 \cdot X_1 \cdot X_3 - 2.35 \cdot X_2 \cdot X_3 - 2.59 \cdot X_1^2 + 1.3 \cdot X_2^2 - 2.87 \cdot X_3^2 \qquad (10)$$

$$M_{w\,RN}(\text{g/mol}) = 45,122.2 + 840.5 \cdot X_1 + 407.88 \cdot X_2 - 1193.38 \cdot X_3 + 108.5 \cdot X_1 \cdot X_2$$
$$- 716.5 \cdot X_1 \cdot X_3 - 415.25 \cdot X_2 \cdot X_3 - 622.73 \cdot X_1^2 - 191.98 \cdot X_2^2 - 1036.97 \cdot X_3^2 \qquad (11)$$

Effect on process variables. *Effect of extraction parameters on pectin yield.* The response surface methodology (RSM) plots (Figs. 1, 2) were used for the analysis of the influence of the independent variables on the pectin characteristics (extraction yield, GalA, DE and M_w). For extraction yield of Fetească Neagră (FN) and Rară Neagră (RN) pectin, the three-dimensional graphics are shown in Figs. 1A–C, 2A–C, respectively. As the results of the ANOVA in Table 3 and 3D graphics present, that all the applied variables highly influenced the pectin yield. Extraction yield had a range between 3.96% (microwave power of 280 W, pH 3 for 90 s) and 9.03% (microwave power of 560 W, pH 2 for 120 s) for FN pectin, while for RN pectin, yield varied between 4.81% and 11.23% for similar extraction conditions.

FN					RN				
Source	Sum of squares	Degree of freedom	Mean square	F-value	Source	Sum of squares	Degree of freedom	Mean square	F-value
(A) Pectin yield (%)									
Model	30.61	9	3.40	12.44**	Model	38.71	9	4.30	7.17*
MP	7.57	1	7.57	27.66**	MP	9.44	1	9.44	15.73**
IT	3.35	1	3.35	12.26*	IT	4.18	1	4.18	6.96*
pH	12.20	1	12.20	44.61**	pH	14.93	1	14.93	24.88**
MP×IT	1.17	1	1.17	4.26ns	MP×IT	0.46	1	0.46	0.77ns
MP×pH	0.70	1	0.70	2.58ns	MP×pH	0.22	1	0.22	0.37ns
IT×pH	1.39	1	1.39	5.09ns	IT×pH	2.07	1	2.07	3.46ns
$R^2 = 0.942$					$R^2 = 0.902$				
(B) GalA (g/100 g)									
Model	1365.79	9	151.75	10.95*	Model	1567.92	9	174.21	11.99*
MP	344.66	1	344.66	24.88**	MP	447.15	1	447.15	30.77**
IT	176.34	1	176.34	12.73*	IT	216.84	1	216.84	14.92*
pH	495.65	1	495.65	35.78**	pH	476.79	1	476.79	32.81**
MP×IT	59.91	1	59.91	4.32ns	MP×IT	74.39	1	74.39	5.12ns
MP×pH	81.81	1	81.81	5.91*	MP×pH	119.03	1	119.03	8.19*
IT×pH	48.30	1	48.30	3.49ns	IT×pH	43.30	1	43.30	2.98ns
$R^2 = 0.934$					$R^2 = 0.939$				
(C) DE (%)									
Model	534.71	9	59.41	15.80**	Model	628.89	9	69.88	18.41**
MP	124.66	1	124.66	33.15**	MP	143.91	1	143.91	37.92**
IT	82.69	1	82.69	21.99*	IT	107.60	1	107.60	28.35**
pH	186.24	1	186.24	49.53**	pH	231.02	1	231.02	60.88***
MP×IT	22.28	1	22.28	5.92*	MP×IT	14.40	1	14.40	3.80ns
MP×pH	35.64	1	35.64	9.48*	MP×pH	39.19	1	39.19	10.33*
IT×pH	15.13	1	15.13	4.02ns	IT×pH	22.04	1	22.04	5.81*
$R^2 = 0.953$					$R^2 = 0.959$				
(D) M_w (g/mol)									
Model	3.57×10^7	9	3.97×10^6	11.18*	Model	2.79×10^7	9	3.10×10^6	9.08*
MP	9.53×10^6	1	9.53×10^6	26.85**	MP	5.65×10^6	1	5.65×10^6	16.51**
IT	1.40×10^6	1	1.40×10^6	3.96ns	IT	1.33×10^6	1	1.33×10^6	3.89ns
pH	1.35×10^7	1	1.35×10^7	38.22**	pH	1.14×10^7	1	1.14×10^7	33.28**
MP×IT	3.74×10^4	1	3.74×10^4	0.10ns	MP×IT	4.71×10^4	1	4.71×10^4	0.13ns
MP×pH	2.58×10^6	1	2.58×10^6	7.27*	MP×pH	2.05×10^6	1	2.05×10^6	6.00*
IT×pH	5.47×10^5	1	5.47×10^5	1.54ns	IT×pH	6.89×10^5	1	6.89×10^5	2.01ns
$R^2 = 0.935$					$R^2 = 0.921$				

Table 3. Analysis of variance (ANOVA) of constructed quadratic model. ns, $p > 0.05$; *$p < 0.01$; **$p < 0.001$; ***$p < 0.0001$; FN, Fetească Neagră; RN, Rară Neagră; GalA, galacturonic acid content; DE, degree of esterification; M_w, molecular weight; MP, microwave power; IT, irradiation time.

The RSM plots showed the interaction effects of pH and microwave power, indicating that lower pH of solution led to higher yield of pectin when microwave power was 560 W. The similar phenomenon was identified when MAE was applied to extract pectin from apple pomace[22], grapefruit[33], navel orange peel[34], sour orange peel[35], papaya peel[36] and banana peels[37]. This can be explained by the fact that during the propagation of the microwave power, the material absorbs the energy which result in enhanced heating of the solvent[13]. Moreover, the heat disrupts the cell walls (CWs) along with the diffusion of relevant compounds out of the material matrix[13]. Thus, the results proved that extraction yield of pectin increased as microwave power was enhanced; when the power level was enhanced (from 280 to 560 W), the solubility of pectin samples was increased with the effect of better extraction.

The second significant factor which influences the extraction yield of pectin during MAE was the treatment time. It was established that pectin yield was increased when the extraction time was enhanced up to 500 s[38,39]. On the other hand, Thirugnanasambandham et al.[40] reported that the highest pectin yield extracted from dragon fruit peels was obtained at 400 W of microwave power, for 120 s at 45 °C and 24 g/mL of solid to liquid ratio. Also, for the extraction of pectin from sour orange peel, Hosseini et al.[41] obtained the following optimal conditions in order to obtain the maximum pectin yield, 700 W of microwave power, for 180 s at pH 1.5. These results correlated well with the experimental data obtained in our study for the highest yield of pectin from grape
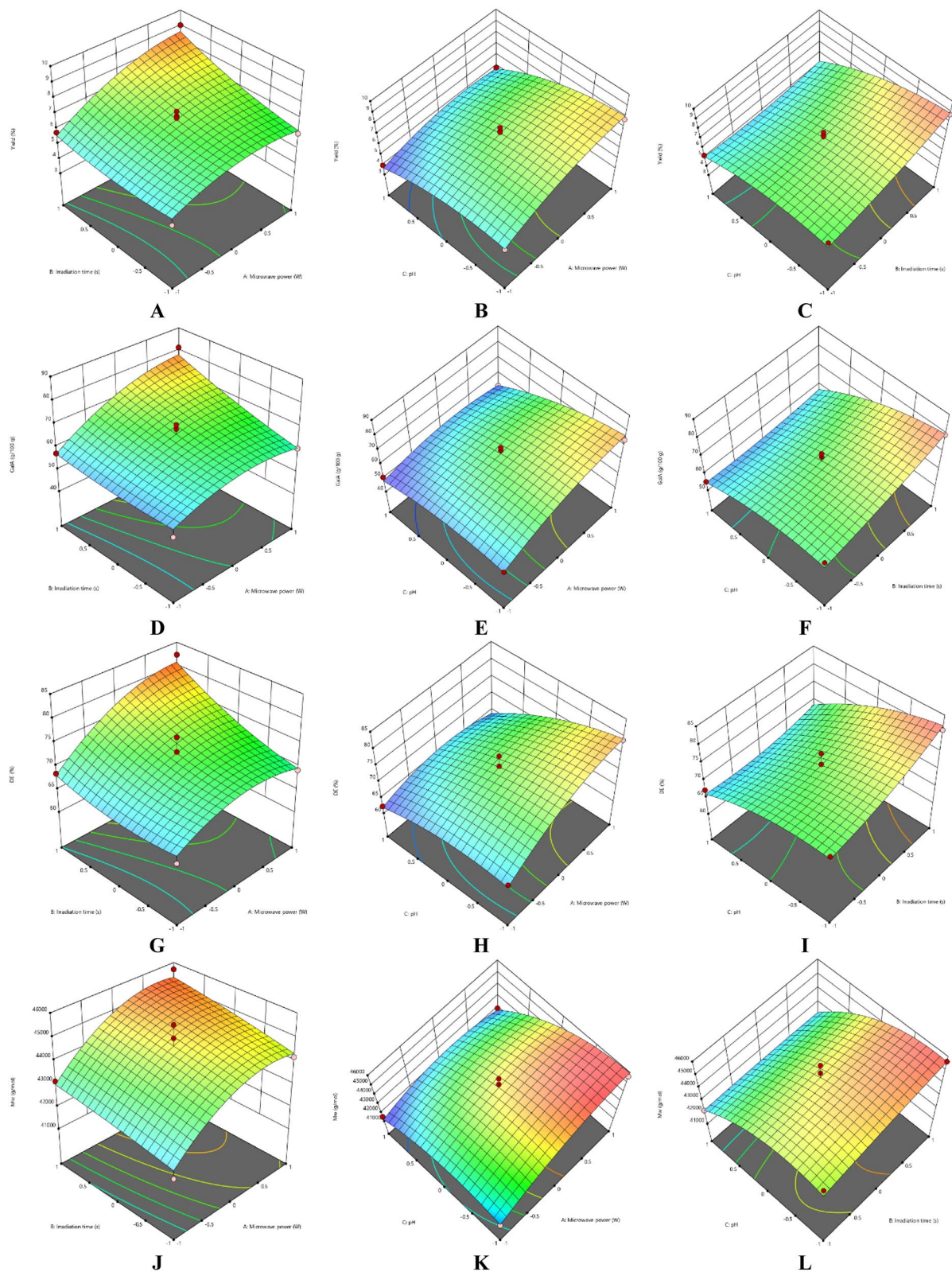
Figure 1. 3D graphs showing the influence of extraction parameters on the yield (**A–C**), GalA content (**D–F**), DE (**G–I**) and M_w (**J–L**) for FN pectin.
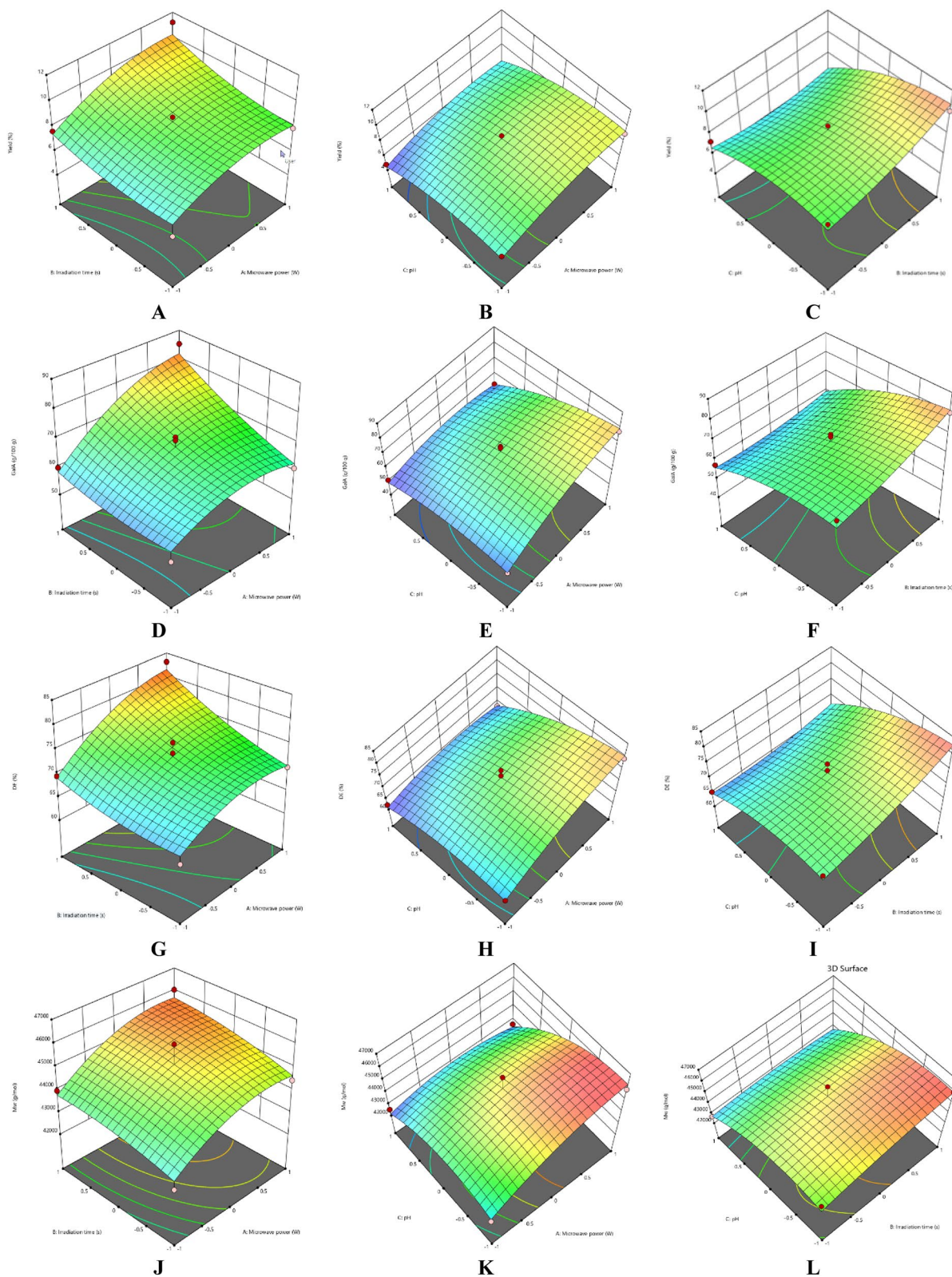
Figure 2. 3D graphs showing the influence of extraction parameters on the yield (**A–C**), GalA content (**D–F**), DE (**G–I**) and M_w (**J–L**) for RN pectin.

pomace (FN and RN) extracted by MAE. Moreover, it could be concluded that MAE was a suitable treatment for extraction of pectin.

Effect of extraction parameters on galacturonic acid content. The influence of extraction characteristics on galacturonic acid (GalA) content of pectin extracted by MAE from FN and RN pomace are shown in Figs. 1D–F and 2D–F, respectively, while the results of the ANOVA are presented in Table 3. In accord with the values in Table 2, the highest GalA content was realized at the correlation of the following parameter values, microwave power of 560 W, pH 2 for 120 s (81.24 g/100 g and 84.18 g/100 g for FN and RN pectin, respectively), while the lowest GalA content (50.92 g/100 g and 51.09 g/100 g for FN and RN pectin, respectively) at the correlation among microwave power of 280 W, pH 3 and 90 s. Figures 1D and 2D indicate that GalA content enhanced with the microwave power, when irradiation time increased. The similar results were obtained when a high microwave power was applied to extract pectin from sour orange peel[35], lime peel[25] and lyophilized pumpkin flesh[42]. Therefore, it can be established that microwave heating may be an effective treatment in order to extract pectin from grape pomace without quality loss.

Figures 1E and 2E present that a higher GalA content were obtained when the pH value was low (less than 2). Thus, the interactions, microwave power-pH and extraction time–pH demonstrated a statistically significant effect on the GalA content of pectin samples extracted by MAE. The same data was achieved by Hosseini et al.[41], they obtained the following optimal conditions: microwave power of 700 W, irradiation time of 180 s and pH 1.5 for the extraction of pectin from sour orange peel. On the other hand, Lefsih et al.[43] reported that a range of pH from 1.5 to 3 didn't affect the GalA content of pectin from *Opuntia ficus indica*. Moreover, the decrease of pH values, the GalA content and yield of pectin were found to decrease[43]. In addition, increasing the irradiation time (Figs. 1F, 2F) enables an enhancement of the GalA content from grape pomace pectin. The enhanced GalA content can be explained by the improved penetration in the plant matrix of microwaves during the extraction[44]. Similar results were reported for the sweet lemon peel pectin extracted by microwave under the optimal conditions (microwave power of 700 W, pH 1.5 and irradiation time of 180 s)[44].

Effect of extraction parameters on degree of esterification. The degree of esterification (DE) is a significant characteristic for the determination of the pectin applications in the food industry, which is related to its texturizing, emulsifying and gelling properties[29,45]. The experimental and predicted values of DE are shown in Table 2, while the ANOVA results for DE are presented in Table 3. The correlations among microwave power, irradiation time and pH which establish the evolution of DE of the grape pomace pectin are illustrated in Figs. 1G–I, 2G–I. According to the results showed in Table 2, all pectin samples had a DE higher than 50%, ranging from 62.28% (microwave power of 280 W, pH 3 for 90 s) to 82.29% (microwave power of 560 W, pH 2 for 120 s) for FN pectin and a range of 62.14–83.11% for RN pectin under similar extraction conditions. The obtained data for DE of the samples presented same tendency in the evolution of the yield and GalA content of pectin.

As can be seen from Figs. 1G–I and 2G–I, the DE of pectin enhanced gradually with the increase of microwave power. At higher microwave power, temperature of the pectin solutions enhanced to improve the diffusion of different compounds. Therefore, the combined influence of irradiation time and microwave power (Figs. 1G, 2G) was more significant than other combined variables presented in Figs. 1I and 2I, as such a higher microwave power needs a shorter extraction duration in order to obtain a high value of DE and vice versa[46]. Some researchers suggest to use the approach of low power with longer irradiation time for extraction as high microwave power presented might reduce purity of pectin[37,46,47]. On the other hand, researchers prefer to use the treatment of high power with short irradiation time for pectin extraction[13,35,44,48].

Effect of extraction parameters on molecular weight. The molecular weight (M_w) of pectin is correlated with its gel-forming, thickening and stabilizing properties, which influence the utilization of pectin in the food industry[49]. Inadequate extraction conditions can affect the structure of pectin and thereby decrease its molecular weight[49,50]. For molecular weight of pectin from FN and RN pomaces, the 3D graphics are shown in Figs. 1J–L and 2J–L, respectively. The M_w of pectin samples ranged from 4.15×10^4 g/mol (microwave power of 280 W, pH 3 for 90 s) to 4.57×10^4 g/mol (microwave power of 560 W, pH 2 for 120 s) and 4.21×10^4 g/mol (microwave power of 420 W, pH 3 for 120 s) to 4.60×10^4 g/mol (microwave power of 560 W, pH 2 for 120 s) for FN and RN pectin, respectively (Table 2).

The microwave power and pH were significantly influenced the M_w values of pectin solutions (Figs. 1K, 2K); this can be explained by that pectin underwent more hydrolysis The same data was obtained by Li et al.[51], they reported the following optimal conditions, 152.63 W of microwave power, pH 1.57 for 3.53 min and 18.92 solid to liquid ratio for sugar beet pulp pectin. Also, they stated that pH of solution had the highest influence on average M_w among the studied variables[51]. Moreover, Yoo et al.[42] noted that pectin extracted by MAE at pH 2 had more than 5 times higher M_w than pectin extracted at pH 1. Microwave power implicates the conversion of high-frequency energy into heat energy which ensues in a more efficient pectin extraction[52]. On the other hand, Bagherian et al.[33] reported that M_w decreased with the increasing of microwave power and heating time; they explained that continued heating may lead to pectin degradation (disaggregation of pectin matrix).

Optimization and validation of extraction conditions. The desirability function approach was utilized to optimize pectin characteristics (extraction yield, GalA content, DE and M_w) concurrently. The first characteristic (y_i) was converted into desirability function (d_i), which varied over the range presented in Eq. (12):

$$0 \leq d_i \leq 1 \tag{12}$$

Sample	Color characteristics			Peak integration		Creep and recovery parameters				
	$L^{\star}$	$C^{\star}_{ab}$	$h^{\star}_{ab}$	ΔH_d (J/g)	T_d (°C)	J_e (1/Pa)	J_r (1/Pa)	$\dot{\gamma}$(1/s)	η (Pa·s)	d(log($\dot{\gamma}$))/d(log(t)) (1/s)
FNP	62.17 (0.05)[a]	12.24 (0.10)[c]	39.10 (0.18)[c]	306.24 (0.12)[a]	276.73 (0.16)[a]	4.547 (0.23)[d]	0.682 (0.21)[c]	0.1252 (0.15)[a]	8.034 (0.27)[d]	0.907 (0.12)[a]
RNP	48.70 (0.02)[b]	17.97 (0.12)[b]	31.66 (0.23)[d]	212.94 (0.09)[b]	267.97 (0.22)[b]	0.993 (0.18)[b]	0.219 (0.16)[b]	0.0564 (0.24)[c]	17.810 (0.32)[b]	0.825 (0.04)[b]
CAP	63.65 (0.06)[a]	20.68 (0.08)[a]	81.18 (0.27)[a]	30.08 (0.17)[c]	217.88 (0.18)[d]	4.334 (0.16)[c]	0.773 (0.08)[d]	0.0902 (0.18)[b]	11.130 (0.24)[c]	0.782 (0.22)[c]
CCP	53.74 (0.11)[b]	12.49 (0.16)[c]	77.14 (0.21)[b]	45.80 (0.24)[c]	238.64 (0.21)[c]	0.037 (0.12)[a]	0.062 (0.07)[a]	0.0004 (0.12)[d]	2380 (0.29)[a]	0.662 (0.08)[d]
F-value	8.81*	47.26***	4692.87***	3.06×10^8***	2.19×10^6***	5.27×10^6***	1.79×10^5***	2.09×10^5***	4.20×10^6***	7.84×10^5***

Table 4. Color characteristics, thermal properties, creep and recovery parameters of pectin samples. Mean values and standard deviation, in brackets. ns, $p > 0.05$, $*p < 0.01$, $**p < 0.001$, $***p < 0.0001$, [a–d]different letters in the same column indicate significant differences among samples ($p < 0.0001$) according to the LSD test with $\alpha = 0.05$. FNP, Fetească Neagră pectin; RNP, Rară Neagră pectin; CAP, commercial apple pectin; CCP, commercial citrus pectin; $L^{\star}$, lightness of the color; $C^{\star}_{ab}$, chroma; $h^{\star}_{ab}$, hue angle; ΔH_d, degradation enthalpy; T_d, degradation temperature; J_e, equilibrium compliance; J_r, recoverable compliance; $\dot{\gamma}$, shear rate; η, viscosity.

In order to achieve the highest extraction yield, GalA content, DE and M_w of pectin from grape pomace (FN and RN), microwave power, irradiation time and pH were optimized. The optimal conditions were the following, 560 W, pH 1.8 and 120 s which presented a desirability function of d = 0.852 and d = 0.861 for RSM plots of FN pectin (9.03% pectin yield, 81.24 g/100 g of GalA content, 82.29% DE and 4.57×10^4 g/mol of M_w) and RN pectin (11.23% pectin yield, 85.18 g/100 g of GalA content, 83.11% DE and 4.60×10^4 g/mol of M_w), respectively. The data was well correlated with the predicted values of responses, so the optimal conditions for MAE of pectin samples were valid.

Color. The pectin color is a significant factor affecting the appearance of gel produced and then the characteristic of the food product in which was added[53]. As can be seen in Table 4, the commercial apple pectin had the highest lightness value (63.65), while the lowest lightness value (48.70) for RN pectin. The samples extracted by MAE, commercial apple and citrus pectin were characterized by more redness. In terms of color, commercial apple and citrus pectin (CAP and CCP) were similar in comparison with FN and RN pectin. Among them, CAP had the highest chroma (20.68) and hue (81.18). Also, the MAE treatment influenced significantly the chroma ($C^{\star}_{ab}$) and hue ($h^{\star}_{ab}$) of pectin samples. This can be explained by the fact that pectin extracted under higher power and temperature for prolonged time has a lower value of lightness ($L^{\star}$). Moreover, the color values of grape pomace pectin were predominantly due to tannins and anthocyanins which are the main polyphenolic compounds responsible for color in red grapes[54]. The similar tendency was reported for pectin extracted from lime peel[25] and apple pomace[55].

FT-IR analysis. The different structural particularities of pectin extracted from grape pomace pectin (FN and RN) by applying microwave technique and correlate them to two commercial pectin samples (apple and citrus), FT-IR analysis was utilized. The FT-IR spectra of commercial samples (apple and citrus pectin) and pectin obtained by MAE in the optimal conditions are presented in Fig. 3. By comparing the spectra, FN and RN pectin had a peak around 3310 cm^{-1} which was ascribed to intermolecular bonding of O(6)H···O(3)[56], while citrus and apple pectin had a shift at 3392–3366 cm^{-1} which was attributed to –OH and carbonyl C=O stretching vibrations[57]. The absorption peaks at 2929–2974 cm^{-1} which were found in pectin samples (commercial and extracted by MAE), was related to –CH (CH, CH$_2$ and CH$_3$) vibrations[58,59]. The C–H stretching detected at the peak of 2348 cm^{-1} is characteristic for polysaccharides chains[60], while the peak of 1868 cm^{-1} was corresponded to the symmetric and asymmetric C–O stretching[61]. The band positions at 1714 cm^{-1} (FN and RN pectin) and 1733 cm^{-1} (commercial apple and citrus pectin) were assigned to undissociated carboxylic acid (COOH) and –CO from the group methyl ester (COOCH$_3$)[62]. The band identified at 1606 cm^{-1} was due to the asymmetric stretching vibration of the carboxylate ion (COO–) and C = C ring stretching of phenolic compounds[63], while the peak at 1559 cm^{-1} was ascribed to the valence vibration of C = O bond[64].

The absorption bands at 1399 cm^{-1} and 1261 cm^{-1} indicated enhancement quantity of carboxylates[65] and –CH bending[66], respectively. The FT-IR spectra in the wavenumber between 1300 and 800 cm^{-1} are referred to as the 'fingerprint region' of carbohydrates, which enables the identification of major chemical groups in different polysaccharides[29,67]. Therefore, the peaks at 1226 cm^{-1}, 1223 cm^{-1} and 1208 cm^{-1} were assigned to the stretching vibration of C single bond, C = O stretching and C–O–H bending, respectively[25]. The peaks at around 1143 cm^{-1} and 1131 cm^{-1} were attributed to the C–O, C–O–C and C–C rings, which are characteristic to the structure of commercial (apple and citrus pectin) samples and pectin (FN and RN) extracted by MAE, respectively[68]. The bands identified at 1072 cm^{-1} (commercial apple and citrus pectin) and 1065 cm^{-1} (FN and RN pectin) were ascribed to C–O and C–C stretching of xyloglucan[69] and galactoglucomannan[68], respectively. Some peaks are more intense in isolated pectin, such as at 1012 cm^{-1} (C–O, C–C and C–O–H stretching)[69], 842 cm^{-1} (CH$_2$ bending linked to α-arabinose pyranoid ring)[68], 828 cm^{-1} and 830 cm^{-1} (α-D-mannopyranose)[70].

Thermal analysis. The DSC was employed to examine the thermal characteristics and describe changes during thermal denaturation of pectin samples (FN and RN pectin extracted by MAE, commercial apple and citrus pectin) as illustrated in Fig. 4 and Table 4. Generally, thermal properties of pectin depend on the inter-
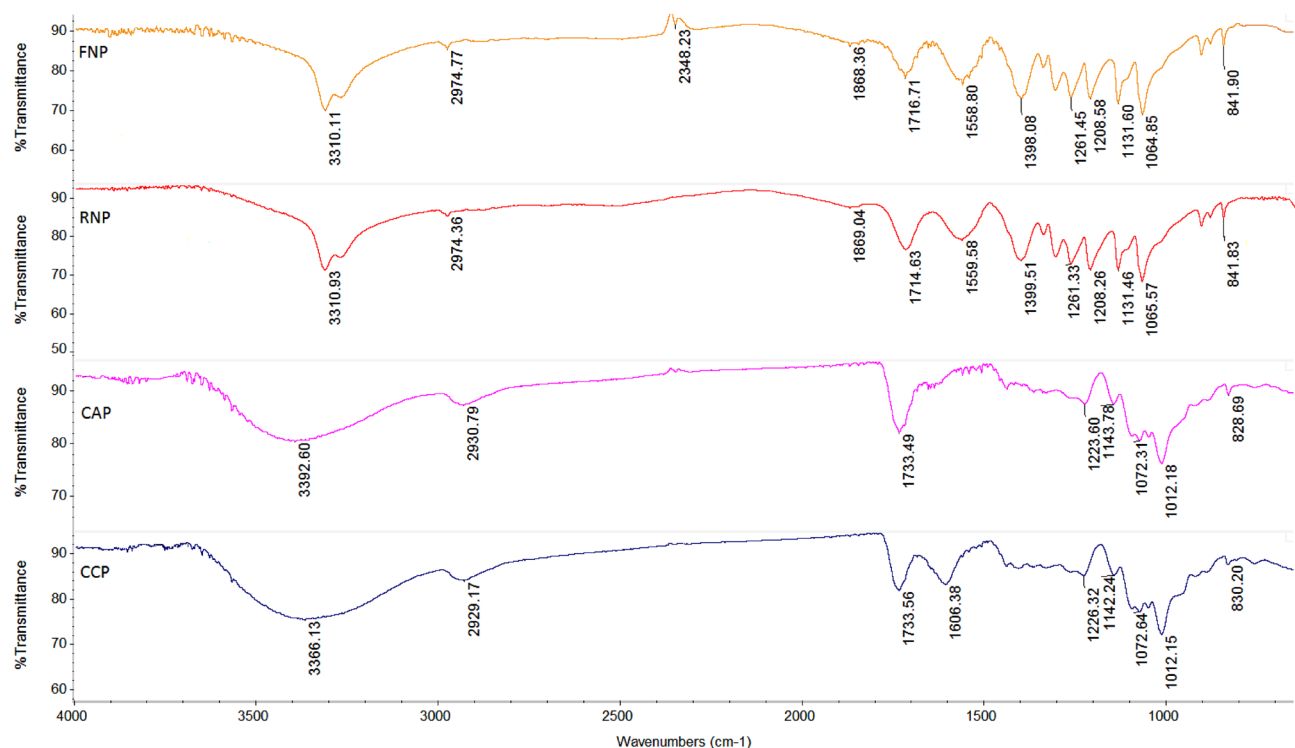
Figure 3. FT-IR spectra of pectin extracted from grape pomace (FN and RN) by MAE under the optimal conditions, commercial apple and citrus pectin (CAP and CCP).
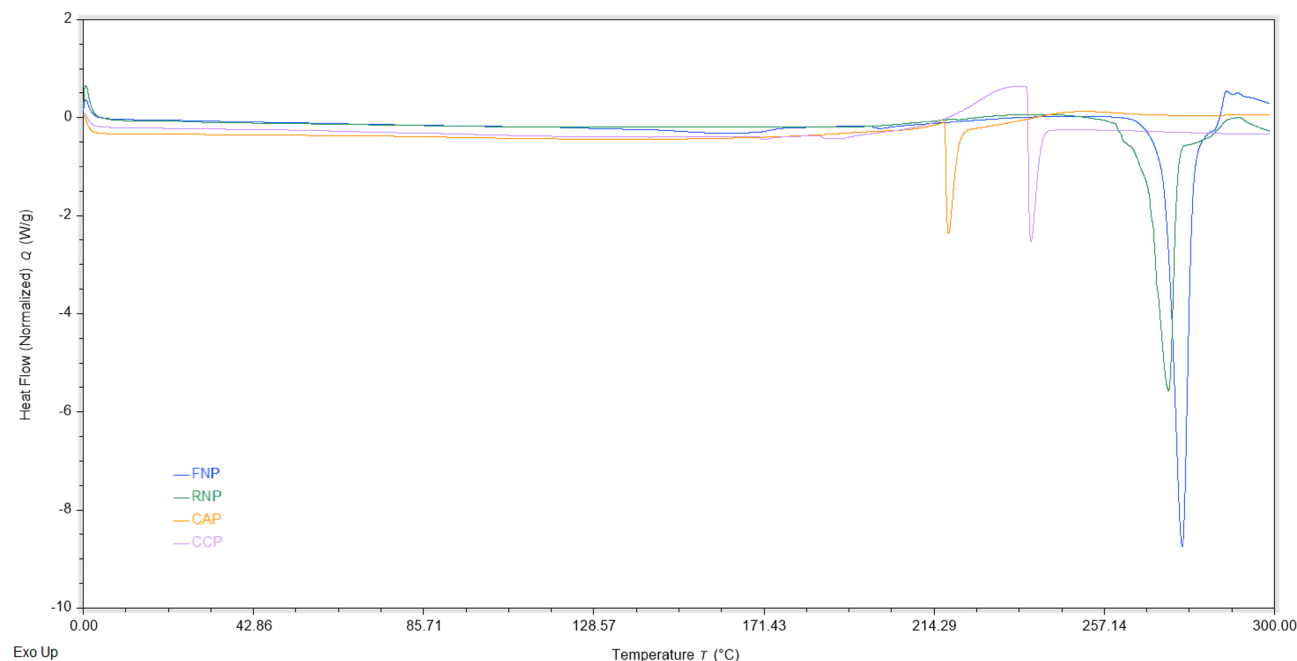

Figure 4. DSC thermograms of pectin extracted from grape pomace (FN and RN) by MAE under the optimal conditions, commercial apple and citrus pectin (CAP and CCP).

dependence of three factors, its state transition occurred during processing, chemical composition and stability properties[71,72]. In the thermogram of pectin samples, exothermic peaks (degradation temperature) were registered at temperatures between 217.88 and 276.73 °C, while endothermic peaks (melting temperature) were not noticed. The endothermic peaks appear from water evaporation, melting-recrystallization of the crystallites, hydrogen bonding of GalA units, and also a conformational change of the pectin pyranose rings[72–76]. In this study, the absence of endothermic peaks indicates that no bound water was removed from the pectin samples[18].
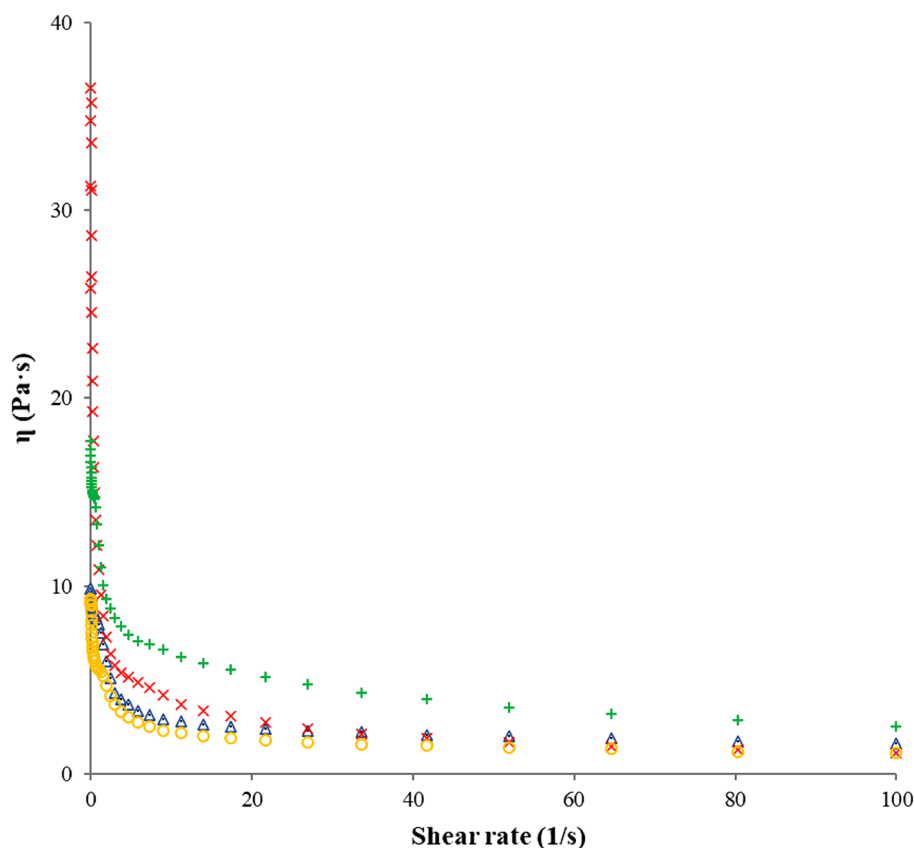
Figure 5. Flow curves of pectin solutions: Fetească Neagră pectin (○), Rară Neagră pectin (+), commercial apple pectin (Δ) and commercial citrus pectin (×); η—dynamic viscosity, $\dot{\gamma}$—shear rate.

Commercial apple and citrus pectin samples had peak temperatures (T_d) at 217.88 and 238.64 °C, respectively, and these results were identical to others established in the scientific studies for the same pectin sources[18,74,75]. The exothermic peaks for pectin samples extracted by MAE had following values: FN—276.73 °C and RN—267.97 °C with a degradation enthalpy (ΔH_d) of 306.24 J/g and 212.94 J/g, respectively. As it can be deduced, pectin obtained by MAE presented higher thermal stability than the commercial sources of pectin, which denotes that FN and RN samples might be preferred during thermal procedure. Therefore, T_d of pectin samples was influenced by their composition, while ΔH_d was affected by GalA content[75].

Rheological properties. *Flow behavior of pectin solutions.* The flow curves of the commercial pectin samples (apple and citrus) and pectin extracted by MAE (FN and RN) are presented in Fig. 5; all curves show a non-Newtonian fluid behavior with an enhancement in the shear stress and a decrease of the dynamic viscosity. This behavior was assigned to the decrease of the pectin intermolecular forces during the stress application[25,77]. It was noticed that RN pectin extracted by MAE had a higher dynamic viscosity than other samples, which means that source of pectin and different extraction parameters affect the pectin flow behavior[25,29]. The viscosity of RN pectin extracted by MAE at a shear rate of 100 s⁻¹ was 2.53 Pa·s which was higher than viscosity of other samples, commercial citrus pectin (1.61 Pa·s), commercial apple pectin (1.11 Pa·s) and FN pectin extracted by MAE (1.01 Pa·s). Moreover, this value was also higher than the dynamic viscosity obtained at the same shear rate (100 s⁻¹) for different concentration (0.5%, 1%, 2% and 3%) of lime peel pectin solution (less than 1 Pa·s)[25], 1.5% and 2% pectin solutions of sour orange peel (less than 0.01 Pa·s)[44] and 30 g/L pectin solution of finger citron pomace (0.5 Pa·s)[70].

The viscosity of pectin solutions enhances with the increasing pectin concentration, while the intermolecular space between the pectin molecules reduce[78]; this behavior has been noticed for pectin from apple pomace[29,79], citrus peel[80], pomelo peels[46], cacao pod husks[81] and sour orange peel[41].

Viscoelastic properties of pectin solutions. Figure 6 shows the viscoelastic characteristics of the 5% pectin solutions; the elastic modulus (G′) and loss modulus (G″) were determined in the linear viscoelastic region. The elastic modulus (G′) serves as the elastic constituent of the stress, while the loss modulus (G″) determines the energy lost via viscous flow[82]. All pectin samples had a higher G″ (liquid behavior) than G′ (solid behavior) in the 0.1–100 Hz frequency domain applied (Fig. 6). The values of both moduli enhanced proportionally with the frequency. Therefore, the G′ enhances more sharply with frequency correlated to the behavior of G″, until the two curves intersect and elastic constituents override the viscous. For that reason, the ability of pectin network
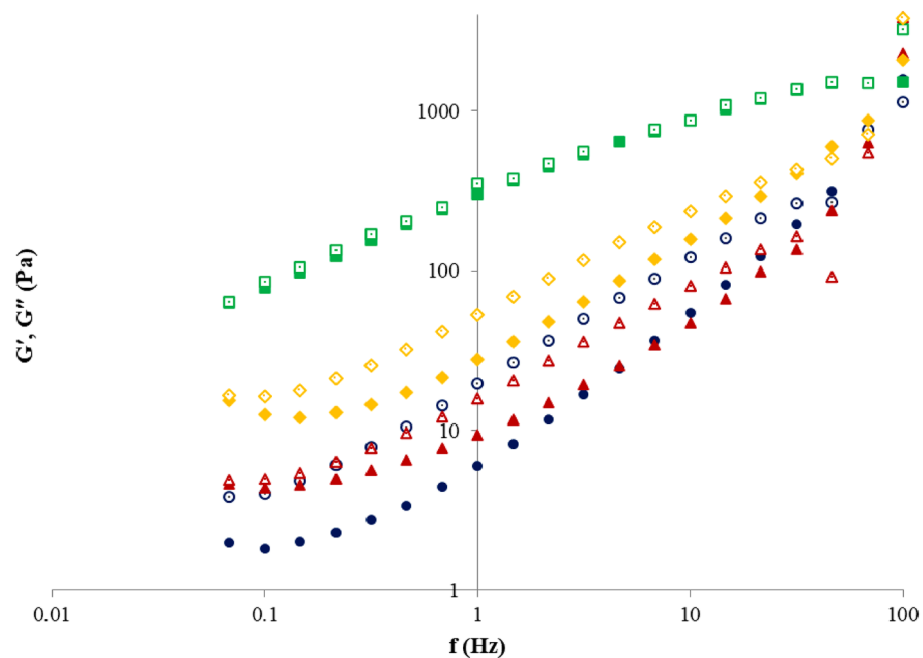
Figure 6. Elastic modulus (fill symbol) and loss modulus (no fill symbol) for different pectin samples: Fetească Neagră pectin (▲, Δ), Rară Neagră pectin (♦, ◊), commercial apple pectin (●, ○) and commercial citrus pectin (■, □); G'—elastic modulus, G''—loss modulus, f—frequency.
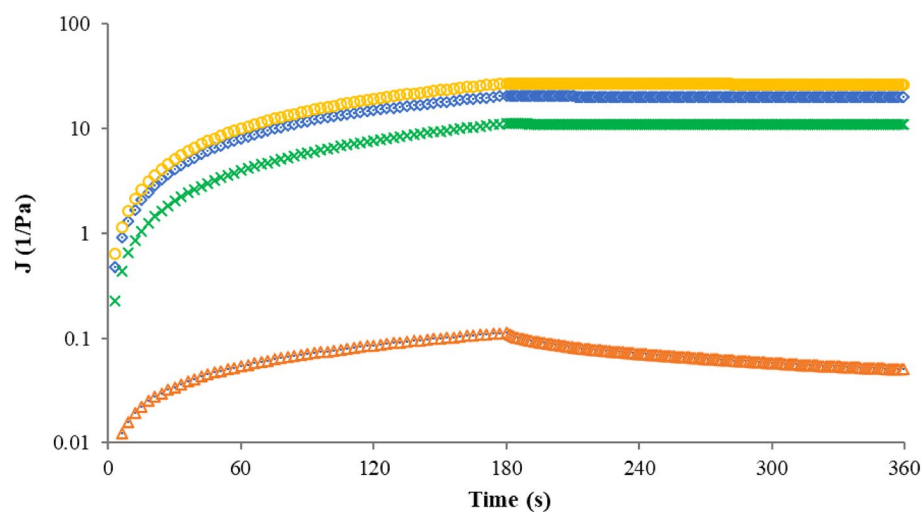

Figure 7. Creep and recovery test of pectin solutions: Fetească Neagră pectin (○), Rară Neagră pectin (×), commercial apple pectin (◊) and commercial citrus pectin (Δ); J—compliance.

to keep the temporarily enforced energy enhances, and it involves more like an elastic solid[81]. Furthermore, the intersection of moduli (G′ and G″) shows the good viscoelasticity of pectin solutions[83]; the lower the value of intersection moment, the major the role of elasticity[84]. The similar behavior was also noticed for the 5% pectin solutions from cacao pod husks[81], lime peel waste[25], pulp of gabiroba[85] and apple pomace[79]. In addition, the extraction methods had a considerable impact on the rheological parameters of pectin and samples extracted by microwave treatment could be considered adequate for using in diverse food products.

'Creep and recovery' analysis. In the 'creep and recovery' analysis (Fig. 7), the pectin samples were subjected to a constant stress during 360 s in order to assess the material deformation; the 'creep' test is from 0 to 180 s, while the 'recovery' test is from 180 to 360 s. The 'recovery' analysis allows access to the rheological behavior of material and lower shear rates for various systems[86]. The 'creep and recovery' analysis, through the creep compliance (J) as time function, distinguishes between the viscous and elastic phases. In viscoelastic materials, 'recovery' phase of the applied stress is partial, controlled by viscous or elastic characteristic of the samples, established at a transitional point between liquid and solid[86]. All samples (FN and RN pectin extracted by MAE; commercial apple and citrus pectin) manifested a non-Newtonian behavior, with a decrease of strain response during the applied stress, evidencing their viscoelastic characters. Commercial citrus pectin solution had a better recovery than other pectin samples. The creep and recovery parameters are presented in Table 4; the equilibrium and recoverable compliance (J_e and J_r), respectively) values for commercial citrus pectin and RN pectin were lower than commercial apple pectin and FN pectin. The highest shear rate ($\dot{\gamma}$) was observed for FN pectin (0.1252 1/s), while the lowest was obtained for CCP (0.0004 1/s); the similar tendency was noticed for d(log($\dot{\gamma}$)/d(log(t)).

Microstructure. The structural morphology of the pectin extracted by MAE from grape pomace (FN and RN) and commercial pectin samples (apple and citrus) was analysed by scanning electron microscopy (SEM). As Fig. 8 shows, the FN and RN pectin samples obtained by MAE appeared to be very different from commercial apple and citrus pectin, having a coarse and slightly ruptured surface. The structure appeared to be influenced by the accelerated enhancement of temperature and the high internal pressure associated with MAE method[46,87]; similar results in terms of morphology were obtained by Liew et al.[46]. Moreover, microwave irradiation causes a great disintegration in the structural morphology of the raw material, which generates an increase of pectin yield[55,87]. The structure of commercial citrus pectin and grape pomace pectin (FN and RN) was found to have a large number of irregular particles with a rough surface (Fig. 8). This may due to the fact that citrus and grape pomace pectin are rich in insoluble fibres (e.g., lignin, cellulose and hemicellulose); similar structural morphologies have been noted in dried pomace from different vegetables and fruits[25,88]. There are some precise differences in the structure of the samples; the commercial apple pectin showed a slight tendency to curl, while the citrus and grape pomace pectin samples seemed to be ruptured. It was also noticed a more homogenous distribution of particle sizes, an increasing number and size of cavities in the structure of the MAE samples (FN and RN pectin), which could be due to pressure rise and sharp intracellular temperature[55]. From the obtained results, it can be concluded that MAE technique influenced the surface morphology of pectin samples extracted from grape pomace.

Conclusions

Pectin was extracted from grape pomace pectin (FN and RN) by MAE using three independent variables, each at three levels, microwave power (280, 420 and 560 W), irradiation time (60, 90 and 120 s) and pH (1, 2 and 3). The microwave power applied for pectin extraction and pH of solution were found to have a high impact on all four responses (extraction yield, GalA content, DE and M_w), while irradiation time had a great influence on pectin yield, GalA content and DE. The optimal conditions for pectin extraction were 560 W, pH of 1.8 for 120 s. The pectin sample extracted by MAE under optimal conditions were compared to CAP and CCP by FT-IR, rheological analysis, DSC and SEM. The viscosity of RN pectin extracted by MAE had a higher viscosity than viscosity of other samples. The microstructure of the pectin samples appeared to be very different from commercial apple and citrus pectin. The grape pomace was found to be a relevant and unconventional source of pectin with a high GalA content, DE and M_w. The physicochemical properties, morphological characteristics and rheological behavior of pectin extracted by MAE from grape pomace denoted a promising field of different applications of this fiber in food industry.
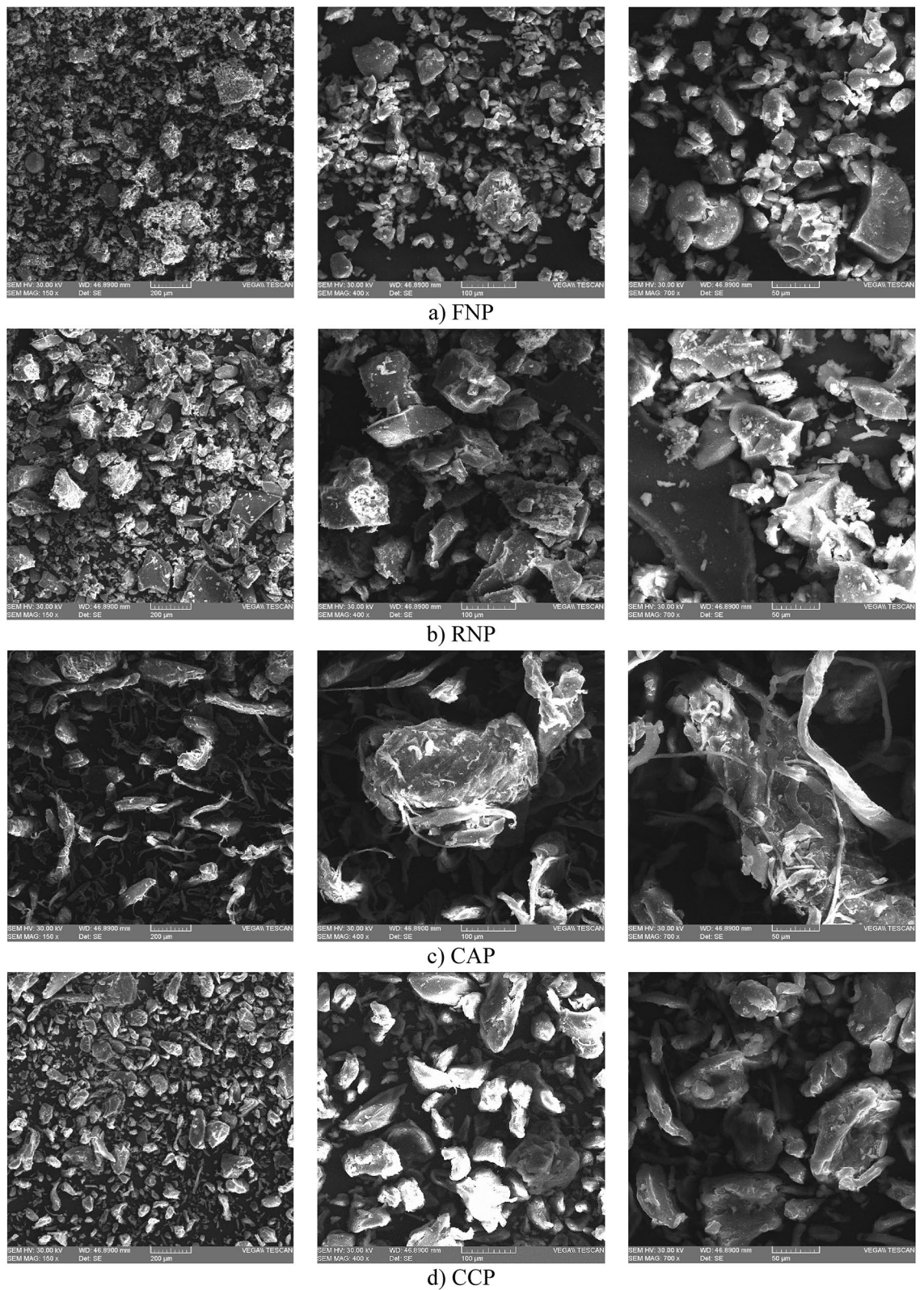
Figure 8. SEM images of pectin solutions: Fetească Neagră pectin (FNP), Rară Neagră pectin (RNP), commercial apple and citrus pectin (CAP and CCP).

Data availability

The datasets used and/or analyzed during the current study available from the corresponding author on reasonable request.

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Acknowledgements

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Competing interests

The authors declare no competing interests.

Additional information

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