

Supplementary Materials

Waste-to-Worth: Nano activated carbon from fruit peels for sulfur-free sugarcane juice clarification

Ramesh Duraisamy^a, Wabi Regasa Bogale^{a,b} and Gada Muleta Fanta^{a,c,d,*}

^aDepartment of Chemistry, College of Natural and Computational Sciences, Arba Minch University, P.O. Box 21, Arba Minch, Ethiopia.

^bDepartment of Industrial Chemistry, College of Natural and Computational Sciences, Bule Hora University, P.O. Box 144, Bule Hora, Ethiopia.

^cDepartment of Materials Science and Engineering, Adama Science and Technology University, P.O. Box 1888, Adama, Ethiopia.

^dCenter of Advanced Materials Science and Engineering, Adama Science and Technology University, P.O. Box 1888, Adama, Ethiopia.

* Corresponding author: gada.muleta@astu.edu.et (G.M. Fanta)

Optimisation procedure of the mixing proportion of fruit peels

As seen in Table S1, the quadratic model of the central composite design (CCD) methodology of three factors of response surface (RSM) has been used to determine the best combination of independent variables (parts of avocado, banana, and papaya fruit peels mixed, total amount of mixed peels kept at 100 g). Design-expert software (v. 13.0.5.0, USA) was used, and the data was analyzed using the RSM approach. In this study, the number of peels (100 g) of avocado (A), banana (B), and papaya (C) peels, with various ratios (1 to 3), was selected as the independent variable for the experiment. Carbon yield (%) was used as the response variable to optimize the combination of independent variables.

As shown in the experimental design in Table S1, peel waste was added and homogenized, yielding a high yield of mixed peel waste. Then, a better peel ratio was optimized to achieve a higher carbon yield using Design-Expert software v. 13.0.5.0. The optimized peel ratio was used for activation and carbonization to synthesized nano activated carbon (NAC).

Table S1. Experimental design for optimising the fruit peel powder mixing proportion and the nano activated carbon preparation.

Run	Independent variables to optimize the proportions of fruit peel (parts)			Independent variables to optimize the nano activated carbon preparation		
	A: Avocado peel	B: Banana peel	C: Papaya peel	A: Impregnation ratio	B: Temperature	C: Time
1	27.03	27.03	45.94	33.3:66.7	700	100
2	14.3	42.85	42.85	33.3:66.7	400	100
3	42.85	42.85	14.3	50:50	700	40
4	33.33	33.33	33.34	50:50	400	70
5	42.85	14.3	42.85	25:75	550	70
6	43.5	43.5	13.0	40:60	550	70
7	27.03	45.94	27.03	25:75	338	70
8	45.94	27.03	27.03	25:75	762	28
9	43.5	13.0	43.5	25:75	550	112
10	33.33	33.33	33.34	25:75	550	70
11	33.33	33.33	33.34	25:75	550	70
12	13.0	43.5	43.5	25:75	550	100

Triplicate experiments (n=3) were performed.

Experimental design for optimisation of cane juice clarification using NAC

The experimental design with two levels of each factor yields 23 runs (including 21 basic runs and 2 replicate central points). Independent variables and their levels were used as shown in Table 3.3: NAC concentration (A: 0.002 - 0.02 %), pH (B: 4 - 8) of juice, clarification time (C: 20 - 100 minutes), clarification temperature (D: 40 – 90 °C) and Agitation speed (100 - 250 rpm). The clarification conditions for optimization levels are selected based on preliminary experiments and literature (Borhan, 2015; Mohammad-Khah and Ansari, 2009). The selected dependent variables used, as shown in Table S2, are turbidity (NTU), mud volume (mL), settling rate, and clarification efficiency (%).

Table S2. Experimental design for the clarification of cane juice for raw sugar production

Run	Independent variables					Dependent variables (responses)			
	A	B	C	D	E	Turbidity (NTU)	Settling rate (cm/minute)	Mud volume (mL)	Color removal efficiency (%)
1	0.02	8	20	90	100				
2	0.027	6	60	65	200				
3	0.011	9.6	60	65	200				
4	0.011	6	60	19	200				
5	0.02	8	20	40	250				
6	0.011	6	60	65	200				
7	0.011	6	60	65	200				
8	0.011	6	60	111	200				
9	0.011	6	12.8	65	200				
10	0.002	8	100	40	250				
11	0.002	4	20	40	100				
12	0.005	6	60	65	200				
13	0.02	4	100	90	100				
14	0.02	8	100	40	100				
15	0.002	8	100	90	100				
16	0.002	4	100	90	175				
17	0.011	6	132.8	65	200				
18	0.011	6	60	65	250				
19	0.011	6	60	65	175				
20	0.02	4	20	90	250				
21	0.002	8	20	90	250				
22	0.02	4	100	40	175				
23	0.011	2.4	60	65	200				

A: Nano activated carbon dose (g); B: pH; C: Time (minutes); D: Temperature (°C); E: Agitation speed (rpm); Triplicate experiments were performed (n=3)

The processing conditions were optimized by lowering turbidity and mud volume, and by increasing other responses (settling rate and clarification efficiency). The regression model was used to generate 3D response plots and contour maps for optimization and to describe the influence of the parameters (independent variables) according to the literature (Mada et al., 2022). Based on the experimental design (shown in Table 3.3), 100 mL of extracted cane juice was taken into a measuring cylinder to set the experimental conditions; then, the respective responses were recorded. In this study, each experimental run was performed in triplicate, and the result is presented as mean \pm SD. Results were analyzed using the RSM-CCD quadratic model in Design-Expert software (v. 13.0.5.0, USA).

Table S3. ANOVA results for the quadratic model of percent carbon yield obtained from the mixed fruit peels (MFP) under carbonization.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	65.02	9	7.22	512.50	0.0019	Significant
A-Avocado	26.45	1	26.45	1876.23	0.0005	
B-Banana	8.48	1	8.48	601.50	0.0017	
C-papaya	0.4380	1	0.4380	31.08	0.0307	
AB	0.0114	1	0.0114	0.8066	0.4639	
AC	12.56	1	12.56	891.33	0.0011	
BC	0.4657	1	0.4657	33.04	0.0290	
A ²	6.04	1	6.04	428.32	0.0023	
B ²	0.0537	1	0.0537	3.81	0.1901	
C ²	1.07	1	1.07	75.82	0.0129	
Residual	0.0282	2	0.0141			
Lack of Fit	0.0282	1	0.0282	4.11	0.5327	non-significant
Pure Error	5.000E-07	1	5.000E-07			
Cor Total	65.05	11				

Triplicate experiments (n=3) were performed

Table S4. Analysis of variance (ANOVA) results for the fixed carbon contents and surface area on synthesized nano activated carbon (NAC) from mixed fruit peel wastes.

Source	Sum of Squares		df	Mean Square		F-value		p-value		
	FCC	SA		FCC	SA	FCC	SA	FCC	SA	
Model	512.70	1.660E+05	9	56.97	18446.92	1227.41	96.15	0.0008	0.0103	Significant
A: IR	0.0782	424.24	1	0.0782	424.24	1.69	2.21	0.3237	0.2754	
B: Temp.	9.64	874.95	1	9.64	874.95	207.72	4.56	0.0048	0.0162	
C: Time	58.88	12400.33	1	58.88	12400.33	1268.59	64.64	0.0008	0.0151	
AB	203.96	10276.19	1	203.96	10276.19	4394.43	53.56	0.0002	0.0182	
AC	49.62	16101.29	1	49.62	16101.29	1069.15	83.93	0.0009	0.0117	
BC	213.03	25253.29	1	213.03	25253.29	4589.93	131.63	0.0002	0.0075	
A ²	99.41	61484.60	1	99.41	61484.60	2141.94	320.48	0.0005	0.0031	
B ²	8.45	573.13	1	8.45	573.13	182.12	2.99	0.0054	0.2261	
C ²	86.80	9570.22	1	86.80	9570.22	1870.27	49.88	0.0005	0.0195	
Residual	0.0928	383.70	2	0.0464	191.85					
Lack of Fit	0.0928	383.70	1	0.0928	383.70	0.8143		0.3776		Non-significant
Pure Error	0.0000	0.0000	1	0.0000	0.0000					
Cor Total	512.80	1.664E+05	11							

IR: impregnation ratio (A); Temp.: temperature (B); C: time; FCC: fixed carbon content; SA: surface area. Triplicate experiments (n=3) were performed.

Table S5. Results of analysis of variance for the turbidity and settling rate in clarification of cane juice using NAC

Source	Turbidity					Settling rate				
	Sum of Squares	Mean Square	F-value	p-value	T-value	Sum of Squares	Mean Square	F-value	p-value	T-value
Model	1.131E+07	5.655E+05	7660.89	0.0001	860.96	0.4280	0.0214	37614.13	< 0.0001	67.82
A	8.611E+5	8.611E+05	11664.87	< 0.0001	-254.70	0.0824	0.0824	1.448E+05	< 0.0001	49.94
B	1.773E+6	1.773E+06	24019.27	< 0.0001	260.80	0.0648	0.0648	1.139E+05	< 0.0001	-28.81
C	9.280E+5	9.280E+05	12570.46	< 0.0001	-186.05	0.0658	0.0658	1.156E+05	< 0.0001	-35.40
D	1.003E+6	1.003E+06	13590.50	< 0.0001	-189.19	0.0761	0.0761	1.337E+05	< 0.0001	28.53
E	13812.88	13812.88	187.11	0.0053	36.75	0.0020	0.0020	3599.77	0.0003	-0.59
AB	25201.27	25201.27	341.38	0.0029	-41.60	0.0321	0.0321	56360.88	< 0.0001	7.03
AC	2.650E+5	2.650E+05	3589.20	0.0003	138.75	0.0003	0.0003	474.13	0.0021	6.02
AD	1.362E+5	1.362E+05	1845.50	0.0005	100.01	0.0785	0.0785	1.380E+05	< 0.0001	7.05
AE	86223.77	86223.77	1168.01	0.0009	79.82	0.0597	0.0597	1.049E+05	< 0.0001	7.04
BC	2.224E+5	2.224E+05	3012.44	0.0003	-127.51	0.1150	0.1150	2.021E+05	< 0.0001	6.54
BD	5.712E+5	5.712E+05	7737.73	0.0001	-203.41	0.0002	0.0002	357.09	0.0028	4.94
BE	85863.26	85863.26	1163.13	0.0009	-79.23	0.0069	0.0069	12214.52	< 0.0001	4.94
CD	3643.28	3643.28	49.35	0.0197	15.19	0.0362	0.0362	63666.92	< 0.0001	6.55
CE	2730.83	2730.83	36.99	0.0260	13.08	0.0471	0.0471	82783.11	< 0.0001	6.55
DE	6782.83	6782.83	91.88	0.0107	-22.69	0.0081	0.0081	14280.65	< 0.0001	4.94
A ²	2.000E+5	2.000E+05	2709.61	0.0004	120.60	0.0373	0.0373	65524.35	< 0.0001	2.57
B ²	1.172E+5	1.172E+05	1587.00	0.0006	-93.52	0.0003	0.0003	507.05	0.0020	1.10
C ²	1.136E+5	1.136E+05	1539.27	0.0006	88.98	0.0191	0.0191	33613.46	< 0.0001	1.67
D ²	22626.37	22626.37	306.50	0.0032	-41.96	0.0055	0.0055	9596.65	0.0001	1.10
E ²	2.290E+5	2.290E+05	3102.37	0.0003	-130.05	0.0163	0.0163	28712.64	< 0.0001	1.10
Resi. error	147.64	73.82				1.138E-6	5.689E-7			
Lock of fit	0.0000	0.0000	1.29	0.3122		1.138E-6	1.138E-6	0.2232	0.6388	
Pure error	0.0000	0.0000				0.0000	0.0000			
Cor Total	1.131E+7					0.4280				

A: NAC dose; B: pH; C: Time; D: Temperature; E: Agitation speed. Triplicate experiments (n=3) were performed

Table S6. Results of analysis of variance for the mud volume and color removal efficiencies in clarification of cane juice using NAC

Source	Mud volume					Color removal efficiency				
	Sum of Squares	Mean Square	F-value	p-value	T-value	Sum of Squares	Mean Square	F-value	p-value	T-value
Model	36.01	1.80	5062.55	0.0001	68.82	2122.57	106.13	10842.68	< 0.0001	758.14
A	4.68	4.68	13158.91	< 0.0001	9.51	103.14	103.14	10537.12	< 0.0001	88.65
B	7.22	7.22	20301.15	< 0.0001	-12.50	345.32	345.32	35279.74	< 0.0001	-79.93
C	2.72	2.72	7656.79	0.0001	5.14	18.24	18.24	1863.08	0.0005	19.09
D	1.38	1.38	3874.09	0.0003	3.91	231.13	231.13	23613.02	< 0.0001	49.13
E	0.0578	0.0578	162.52	0.0061	-1.27	7.61	7.61	776.97	0.0013	18.36
AB	0.0022	0.0022	6.23	0.1299	-0.07	1.80	1.80	183.95	0.0054	7.30
AC	3.11	3.11	8735.04	0.0001	-9.33	0.0143	0.0143	1.46	0.3506	0.53
AD	0.7026	0.7026	1975.67	0.0005	-4.50	38.57	38.57	3940.18	0.0003	-33.04
AE	0.3998	0.3998	1124.27	0.0009	-3.43	0.5129	0.5129	52.40	0.0186	3.94
BC	0.5096	0.5096	1432.84	0.0007	4.08	29.42	29.42	3005.95	0.0003	28.95
BD	3.58	3.58	10058.90	< 0.0001	10.16	0.3638	0.3638	37.17	0.0259	3.36
BE	1.16	1.16	3256.34	0.0003	5.82	28.41	28.41	2902.54	0.0003	28.57
CD	0.0218	0.0218	61.27	0.0159	0.85	6.94	6.94	709.18	0.0014	-14.16
CE	0.0087	0.0087	24.51	0.0385	-0.47	0.0000	0.0000	0.0044	0.9532	-0.09
DE	0.4610	0.4610	1296.27	0.0008	3.48	15.22	15.22	1554.82	0.0006	-20.74
A ²	1.88	1.88	5298.72	0.0002	-7.48	1.26	1.26	128.55	0.0077	2.57
B ²	2.05	2.05	5753.56	0.0002	7.60	71.33	71.33	7287.69	0.0001	1.10
C ²	0.0005	0.0005	1.39	0.3590	-0.19	11.83	11.83	1208.38	0.0008	1.67
D ²	0.2006	0.2006	563.97	0.0018	2.68	97.68	97.68	9979.67	0.0001	1.10
E ²	1.23	1.23	3459.48	0.0003	5.80	8.95	8.95	914.48	0.0011	1.10
Residual	0.0007	0.0004				0.0196	0.0098			
Lack of Fit	0.0007	0.0007	0.0457	0.8317		0.0196	0.0196	1.59	0.2142	
Pure Error	0.0000	0.0000				0.0000	0.0000			
Cor Total	36.01					2122.59				

A: NAC dose; B: pH; C: Time; D: Temperature; E: Agitation speed; Triplicate experiments (n=3) were performed