Valorization of food waste through controlled oxidation: a case study on densification of onion waste

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Supplementary information

EDX analysis of samples (Method):

R2, after undergoing pulverization and ensuring complete moisture evaporation, was taken directly for analysis. P1 and P2 were collected after oxidation and drying, scraped from the edges to obtain the powdered samples, and stored for further analysis. The obtained powdered samples were used for for energy-dispersive X-ray spectroscopy (EDX). After sputter coating of samples, it was directly analyzed for identification of spectrum of elements.

EDX analysis of samples(discussion):

The energy dispersive X-ray spectroscopy (EDXS) was performed to check the elemental composition of raw onion powder (R2), densified onion sample when NaOCl was used as oxidant (P1), and densified onion sample when H_2O_2 was used as oxidant (P2). Only the P1 sample shows the presence of Na and Cl elements which are generated due to the oxidant NaOCl. These elements are not detected in the other two samples. The EDXS data was used to validate the peaks in P1 sample at 32°, 45°, and 55° that also correspond to NaCl in XRD analysis (Figure 6).

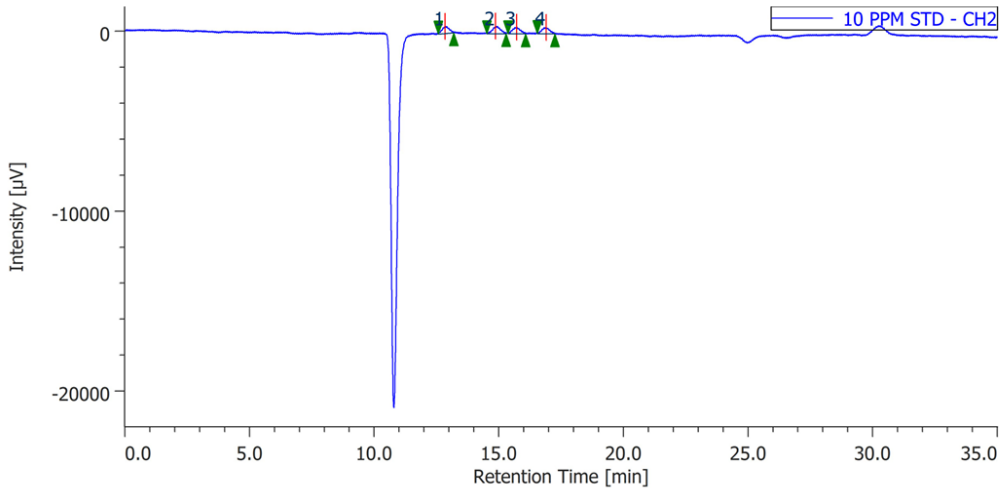
Table 1: EDX analysis of onion powder (R2,) densified onion powder treated with NaOCl (P1) and H2O2 (P2)

Elements	Weight %		
	R2	P1	P2
C	36.7	33.7	35.7

O	57.4	48.0	52.5
Mg	0.8	Not Detected	0.6
S	1.1	1.0	1.7
Cl	0.5	5.4	0.9
K	1.7	3.2	5.9
Ca	1.8	2.8	1.0
Si	Not Detected	0.7	Not Detected
Na	Not Detected	4.3	Not Detected
P	Not Detected	0.9	1.7

HPLC:

1. 10 ppm standard run:



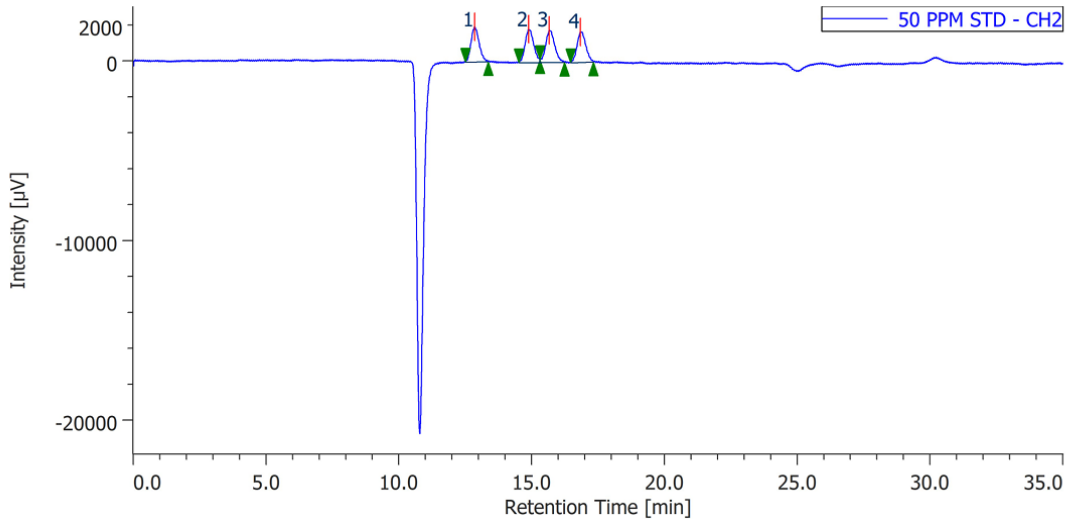
Channel & Peak Information Table

Chromatogram Name	10 PPM STD-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.842	7853	406	23.967	25.979	N/A	8315	3.503	1.129	
2	GLUCOSE	2	14.867	9323	425	28.453	27.228	N/A	9962	1.422	1.103	
3	XYLOSE	2	15.717	7969	369	24.322	23.650	N/A	10882	1.967	1.014	
4	ARABINOSE	2	16.892	7620	362	23.257	23.143	N/A	12895	N/A	0.996	

Figure 1

2. 50ppm standard run:



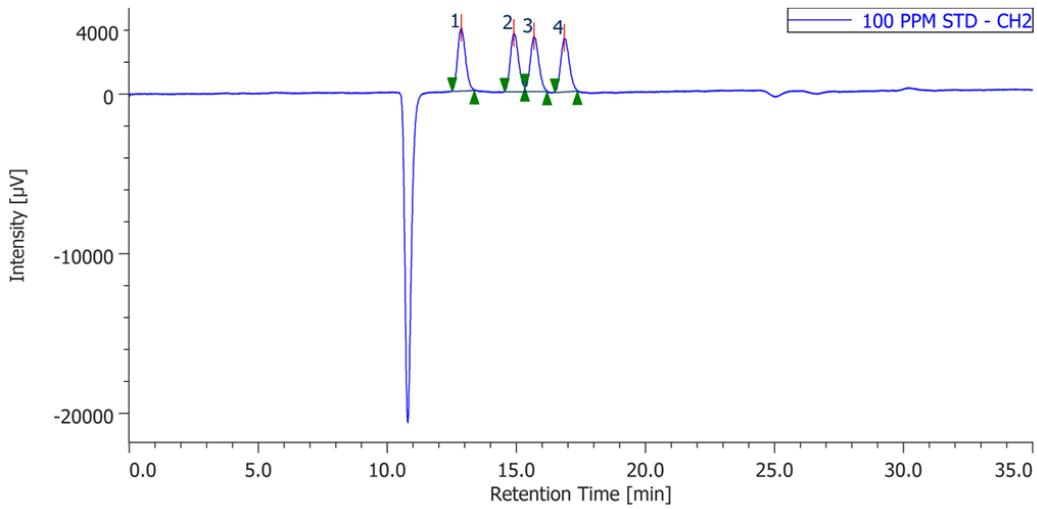
Channel & Peak Information Table

Chromatogram Name	50 PPM STD-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.850	41645	1971	25.200	26.892	N/A	8322	3.499	1.151	
2	GLUCOSE	2	14.883	41910	1872	25.360	25.533	N/A	9787	1.263	N/A	
3	XYLOSE	2	15.658	42560	1794	25.754	24.479	N/A	9909	1.888	N/A	
4	ARABINOSE	2	16.833	39142	1693	23.685	23.096	N/A	11834	N/A	1.201	

Figure 2

3. 100ppm standard run:



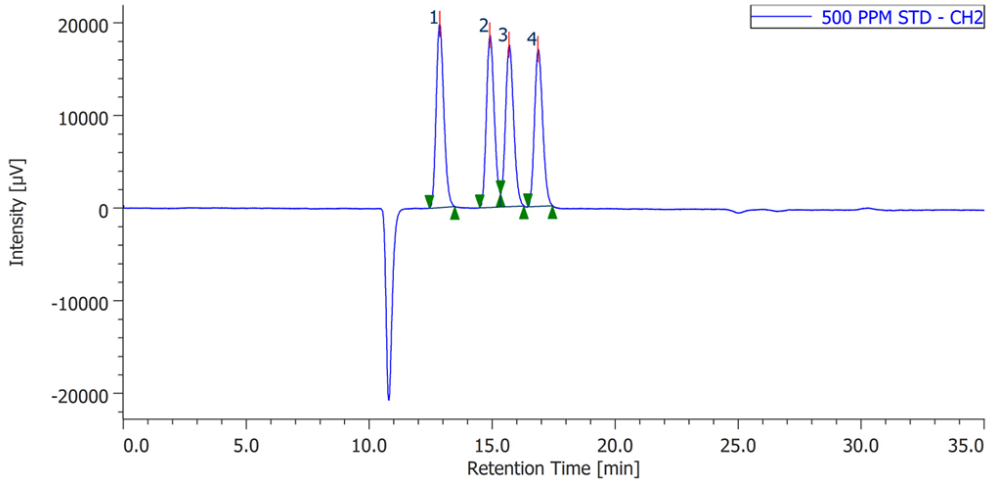
Channel & Peak Information Table

Chromatogram Name	100 PPM STD-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.858	84022	3968	26.106	27.343	N/A	8219	3.531	1.166	
2	GLUCOSE	2	14.892	81666	3700	25.374	25.494	N/A	10278	1.318	N/A	
3	XYLOSE	2	15.675	79231	3474	24.618	23.942	N/A	10772	1.936	N/A	
4	ARABINOSE	2	16.850	76926	3370	23.902	23.221	N/A	12085	N/A	1.196	

Figure 3

4. 500ppm standard run:



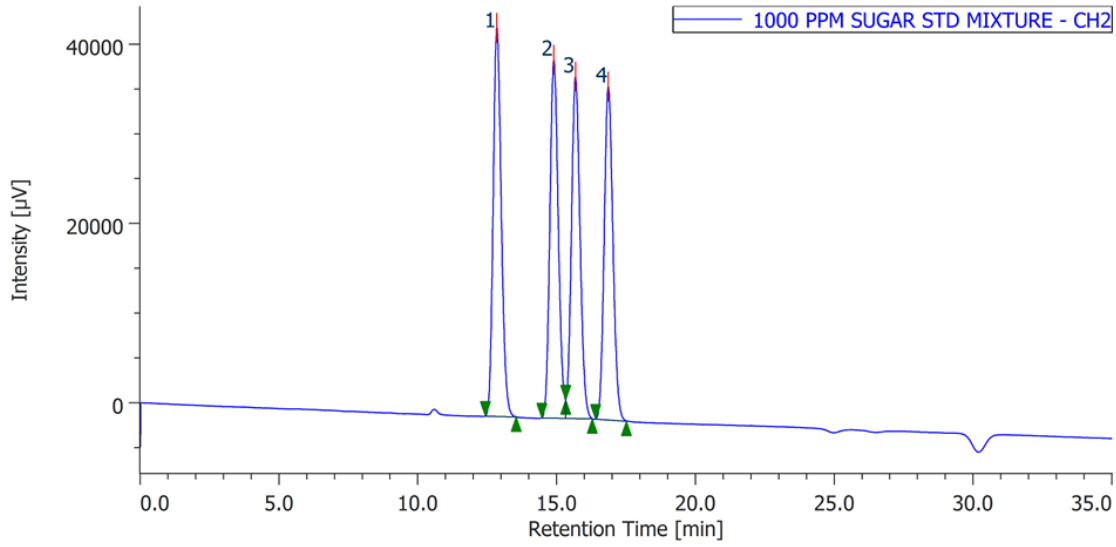
Channel & Peak Information Table

Chromatogram Name	500 PPM STD-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.858	429217	19833	26.245	27.232	N/A	8126	3.522	1.194	
2	GLUCOSE	2	14.900	413632	18559	25.292	25.483	N/A	10142	1.315	N/A	
3	XYLOSE	2	15.683	402813	17473	24.631	23.992	N/A	10837	1.945	N/A	
4	ARABINOSE	2	16.858	389734	16964	23.831	23.293	N/A	12256	N/A	1.190	

Figure 4

5. 1000ppm standard run:



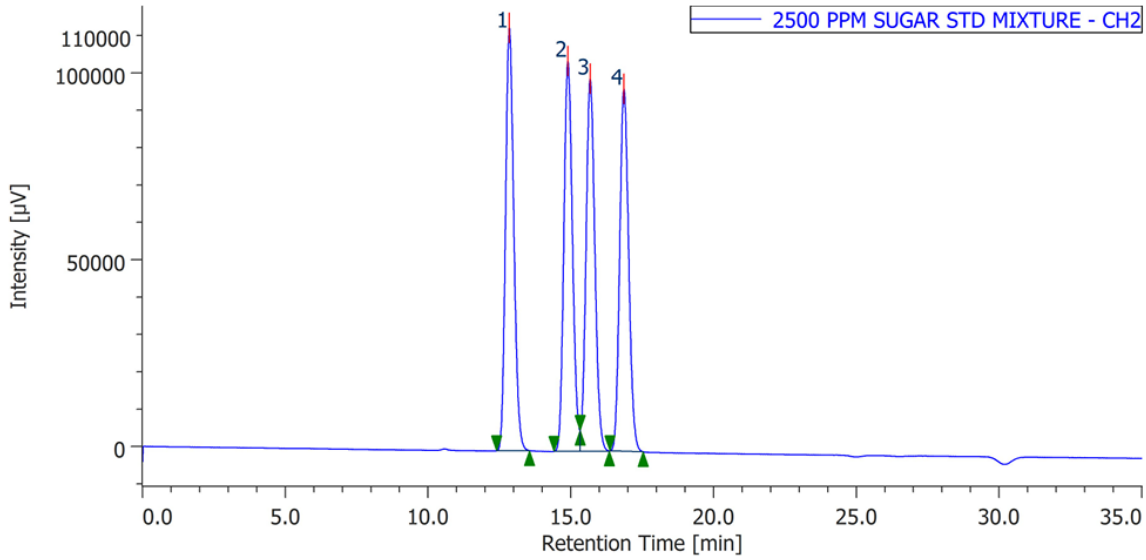
Channel & Peak Information Table

Chromatogram Name	1000 PPM SUGAR STD MIXTURE-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.842	879982	43355	26.242	27.329	N/A	9291	3.770	1.171	
2	GLUCOSE	2	14.892	838570	39973	25.007	25.197	N/A	11414	1.397	N/A	
3	XYLOSE	2	15.675	821564	38138	24.500	24.040	N/A	12237	2.073	N/A	
4	ARABINOSE	2	16.858	813224	37178	24.251	23.435	N/A	13613	N/A	1.114	

Figure 5

6. 2500 ppm standard run:



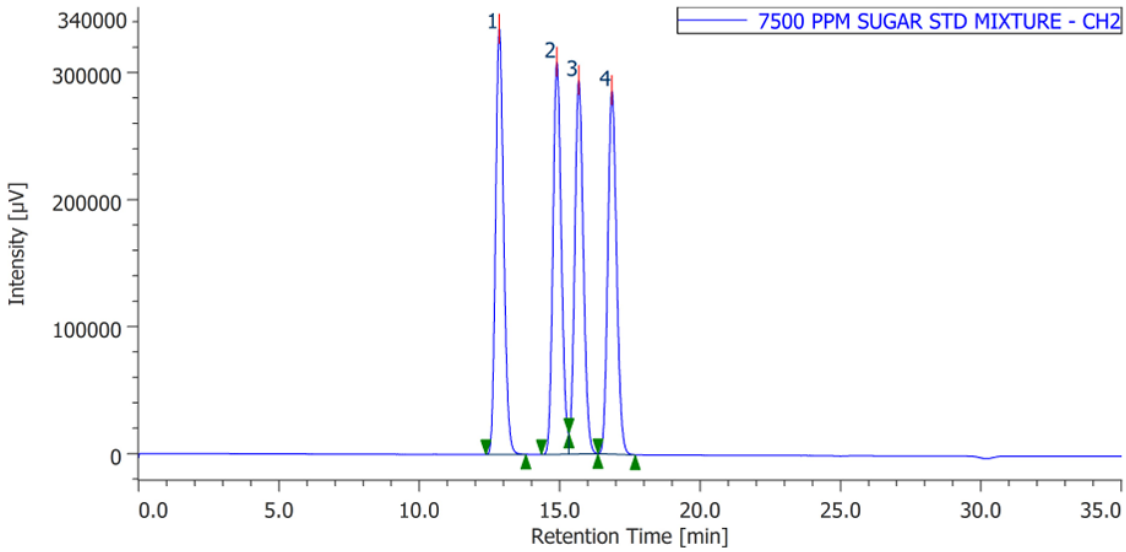
Channel & Peak Information Table

Chromatogram Name	2500 PPM SUGAR STD MIXTURE-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.842	2292291	113185	26.211	27.323	N/A	9293	3.784	1.169	
2	GLUCOSE	2	14.892	2190975	104474	25.053	25.220	N/A	11585	1.403	N/A	
3	XYLOSE	2	15.675	2146240	99632	24.541	24.052	N/A	12270	2.068	N/A	
4	ARABINOSE	2	16.850	2115955	96952	24.195	23.405	N/A	13812	N/A	1.142	

Figure 6

7. 7500 ppm standard run:



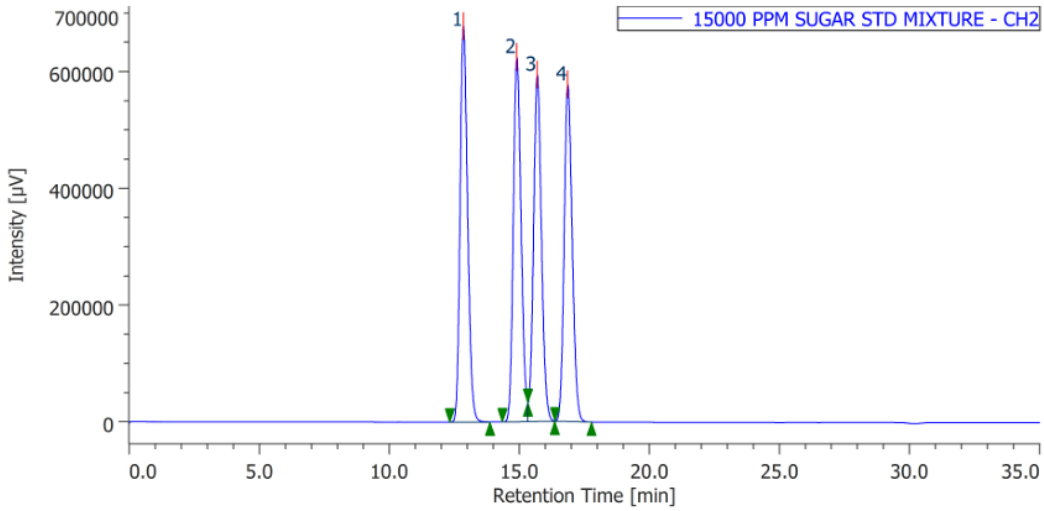
Channel & Peak Information Table

Chromatogram Name	7500 PPM SUGAR STD MIXTURE-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.842	6748728	335148	26.262	27.359	N/A	9466	3.821	1.170	
2	GLUCOSE	2	14.892	6431598	309067	25.028	25.230	N/A	11823	1.415	N/A	
3	XYLOSE	2	15.675	6296874	294376	24.504	24.030	N/A	12440	2.081	N/A	
4	ARABINOSE	2	16.850	6220170	286422	24.205	23.381	N/A	13969	N/A	1.138	

Figure 7

8. 15000 ppm standard run:



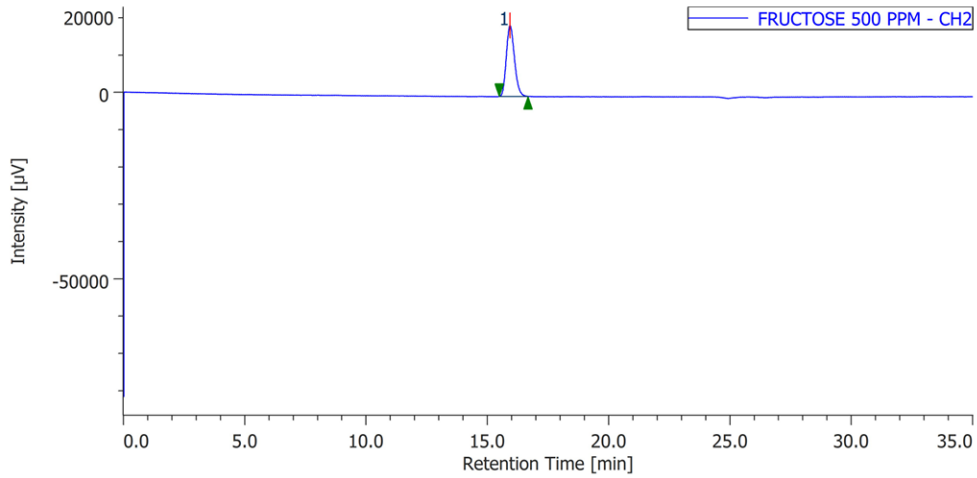
Channel & Peak Information Table

Chromatogram Name	15000 PPM SUGAR STD MIXTURE-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	CELLOBIOSE	2	12.850	13586640	677931	26.309	27.404	N/A	9635	3.851	1.165	
2	GLUCOSE	2	14.900	12922350	624501	25.022	25.244	N/A	12013	1.427	N/A	
3	XYLOSE	2	15.683	12647475	594199	24.490	24.019	N/A	12676	2.095	N/A	
4	ARABINOSE	2	16.858	12486830	577188	24.179	23.332	N/A	14128	N/A	1.141	

Figure 8

9. Fructose standard run:



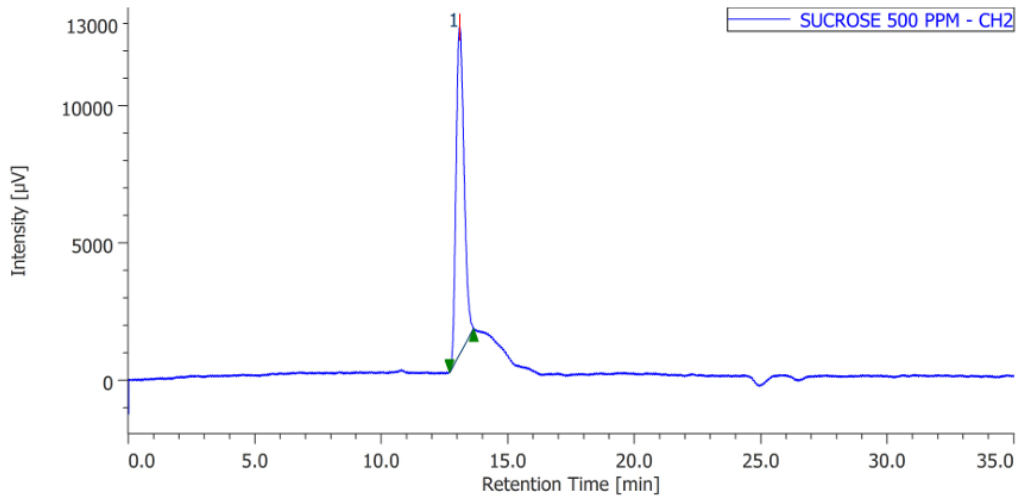
Channel & Peak Information Table

Chromatogram Name	FRUCTOSE 500 PPM-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	FRUCTOSE	2	15.925	446234	19096	100.000	100.000	N/A	10809	N/A	1.218	

Figure 9

10. Sucrose standard run:



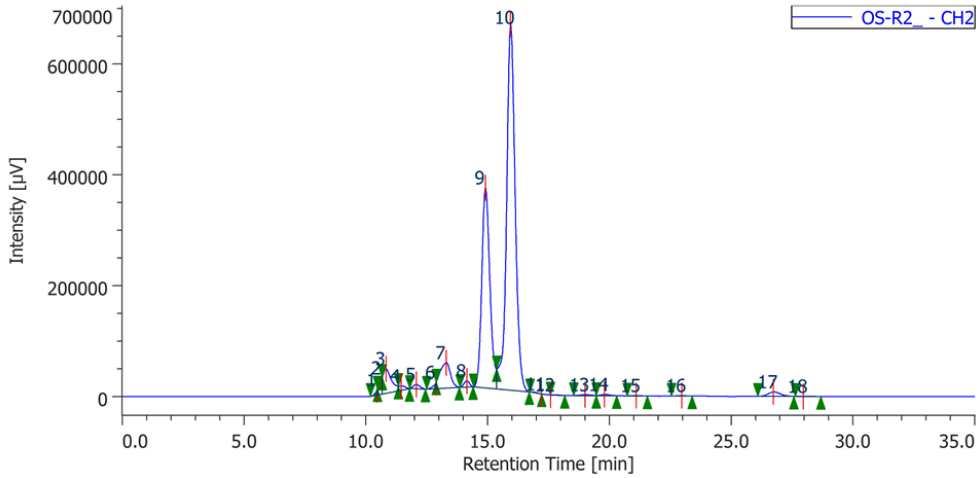
Channel & Peak Information Table

Chromatogram Name	SUCROSE 500 PPM-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	SUCROSE	2	13.100	257341	11900	100.000	100.000	N/A	8308	N/A	1.200	

Figure 10

11. R2(OS for onion sample dried):



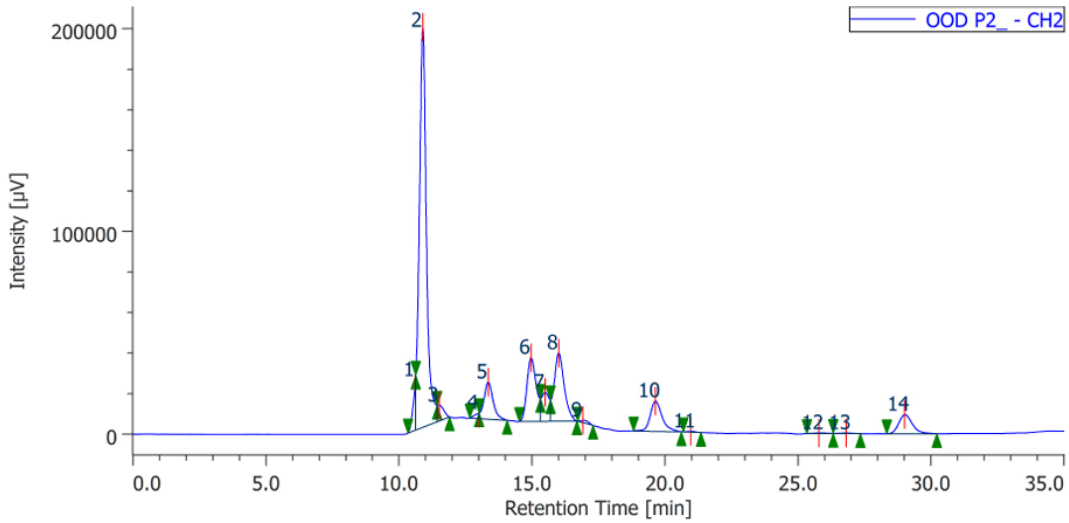
Channel & Peak Information Table

Chromatogram Name	OS-R2_-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	2	10.458	56798	9633	0.201	0.804	N/A	N/A	N/A	N/A	
2	Unknown	2	10.658	240171	28952	0.851	2.416	N/A	N/A	N/A	N/A	
3	Unknown	2	10.825	1043843	43950	3.699	3.668	N/A	N/A	N/A	N/A	
4	Unknown	2	11.450	135155	7588	0.479	0.633	N/A	N/A	N/A	N/A	
5	Unknown	2	12.067	145328	7621	0.515	0.636	N/A	8244	N/A	1.172	
6	CELLOBIOSE	2	12.875	82080	10692	0.291	0.892	N/A	N/A	N/A	N/A	
7	Unknown	2	13.300	1359616	45729	4.817	3.816	N/A	3933	1.299	N/A	
8	Unknown	2	14.142	174528	10875	0.618	0.908	N/A	15724	1.466	0.963	
9	GLUCOSE	2	14.908	8324089	360760	29.495	30.108	N/A	9958	1.694	N/A	
10	XYLOSE	2	15.933	16251378	659258	57.583	55.019	N/A	10708	N/A	N/A	
11	ARABINOSE	2	17.200	43	9	0.000	0.001	N/A	N/A	N/A	N/A	
12	Unknown	2	17.583	5369	13	0.019	0.001	N/A	N/A	N/A	N/A	
13	Unknown	2	19.000	39322	1438	0.139	0.120	N/A	10517	1.133	N/A	
14	Unknown	2	19.783	50822	2139	0.180	0.178	N/A	15063	2.046	N/A	
15	Unknown	2	21.092	16163	692	0.057	0.058	N/A	17511	2.942	1.119	
16	Unknown	2	22.967	20881	866	0.074	0.072	N/A	20569	4.796	0.992	
17	Unknown	2	26.725	273227	7858	0.968	0.656	N/A	13189	1.322	1.139	
18	Unknown	2	27.950	3678	156	0.013	0.013	N/A	14552	N/A	1.651	

Figure 11

12.P2(Oxidized sample-OOD):



Channel & Peak Information Table

Chromatogram Name	OOD P2_-CH2
Sample Name	
Channel Name	CH2
Sampling Interval	500 [msec]
Peak Method	(Manual)

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	2	10.617	163743	24687	2.280	6.935	N/A	N/A	N/A	N/A	
2	Unknown	2	10.892	3634306	197206	50.606	55.396	N/A	9144	N/A	N/A	
3	Unknown	2	11.517	129436	7646	1.802	2.148	N/A	N/A	N/A	N/A	
4	CELLOBIOSE	2	13.000	29350	2661	0.409	0.747	N/A	N/A	N/A	N/A	
5	Unknown	2	13.350	438094	18223	6.100	5.119	N/A	7851	2.541	N/A	
6	GLUCOSE	2	14.958	761441	31315	10.603	8.797	N/A	8060	N/A	N/A	
7	XYLOSE	2	15.492	289915	14194	4.037	3.987	N/A	N/A	N/A	N/A	
8	Unknown	2	16.000	881226	33555	12.271	9.426	N/A	8777	1.417	N/A	
9	ARABINOSE	2	16.908	24194	1067	0.337	0.300	N/A	12622	3.992	1.443	
10	Unknown	2	19.633	465383	14974	6.480	4.206	N/A	10490	2.089	1.281	
11	Unknown	2	20.958	7991	420	0.111	0.118	N/A	27512	6.596	1.106	
12	Unknown	2	25.783	9237	298	0.129	0.084	N/A	11506	1.124	N/A	
13	Unknown	2	26.800	8948	315	0.125	0.088	N/A	15799	2.482	N/A	
14	Unknown	2	29.000	338292	9430	4.711	2.649	N/A	15736	N/A	1.263	

Figure 12

13. The HPLC method was adopted as per NREL Laboratory Analytical Procedure (LAP) protocol with similar mobile phase and calibration standards[1]. After the calibration curve was made, the sample were matched and analyzed.

HPLC analysis of sugars present in the sample

High-performance liquid chromatography (HPLC) analysis was performed using a JASCO CO-4061 system equipped with a refractive index (RI) detector. The separation was achieved with a Shodex SH1011 sugar column (8 mm × 300 mm), coupled with a Shodex SH-G guard column.

The mobile phase consisted of 0.005 M H_2SO_4, delivered at a flow rate of 0.5 mL/min. The column temperature was maintained at 50°C, and the injection volume was 20 µL.In preparation of standard, 4 sugar standards (cellobiose, glucose, xylose, arabinose) were prepared with various concentrations and run on HPLC individually and collectively to make the sugar template and in addition to that, fructose and sucrose were also run as standard (ES Fig 1-7).

In preparation of the sample, 1g of both R2 and P2 were taken in powder form and dissolved in 10 cm^3 of water and sonicated. Then sample solutions were filtered using 0.2 µm filter paper and stored at 8°C. After preparation of the mobile phase, samples were injected for analysis and data were recorded.

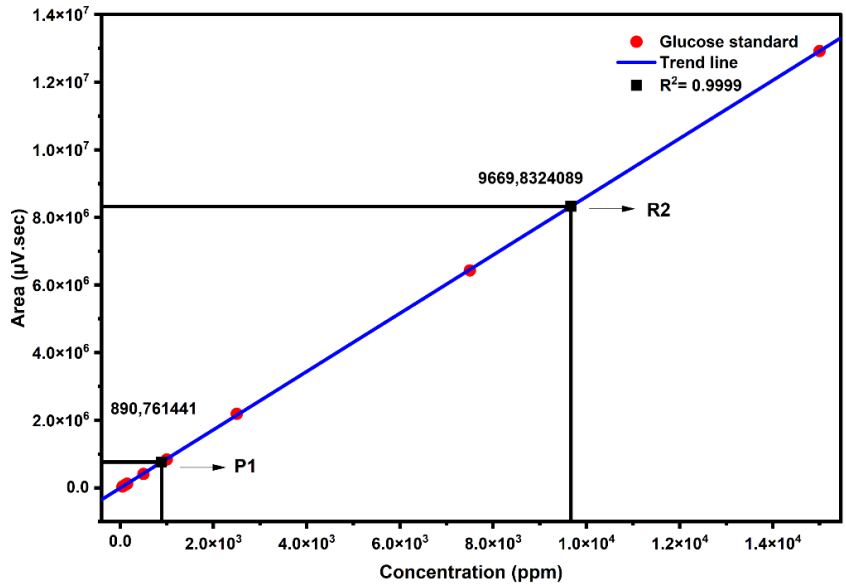


Figure 13: HPLC analisis of waste onion powder (R2) and densified material made from dry onion waste powder using NaOCl (P1) for detection of change in soluble sugar fraction

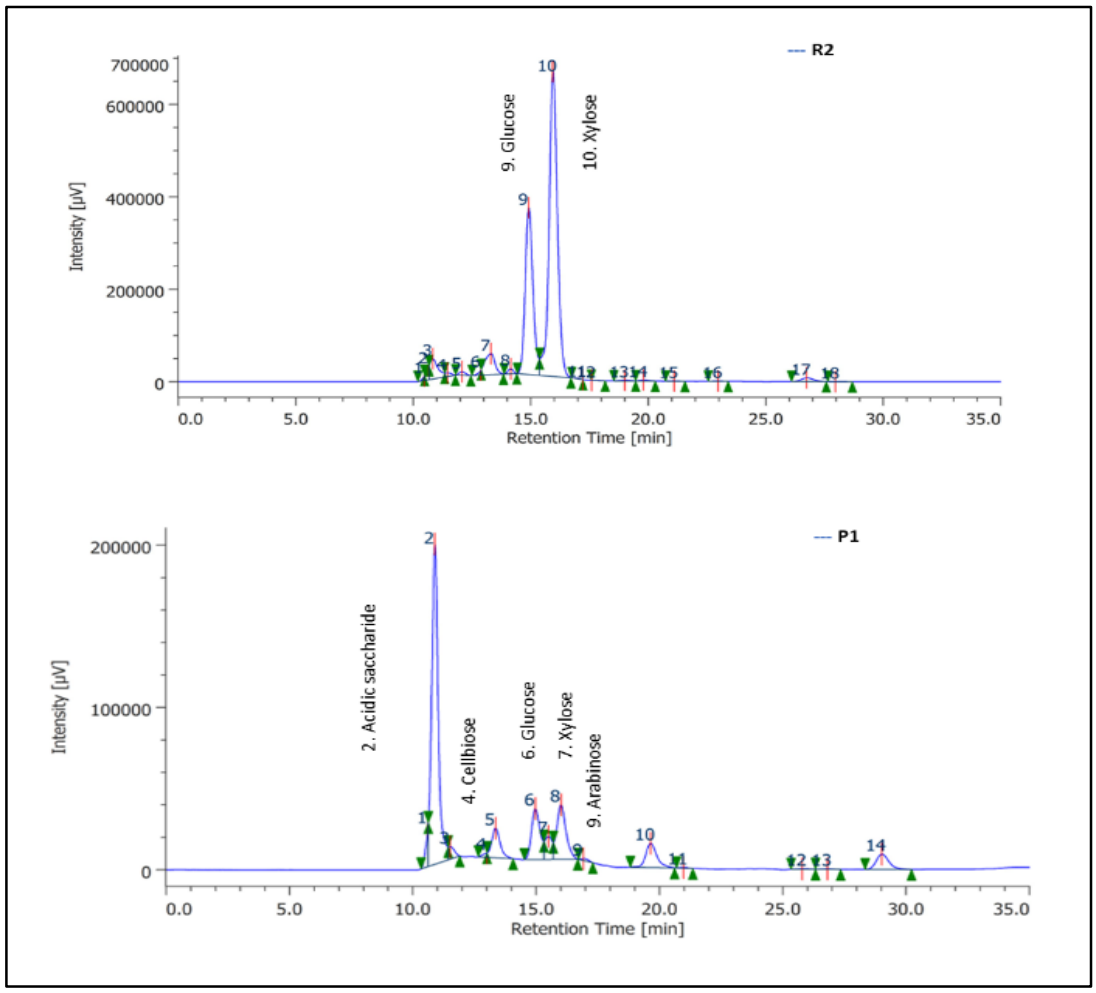


Figure 14: HPLC analysis of waste onion powder (R2) and densified material made from dry onion waste powder using NaOCl (P1) for detection of change in soluble sugar fraction

HPLC analysis was conducted to confirm the oxidation of sugars present in the powdered onion samples (R2). Standards such as glucose, xylose, arabinose, and cellobiose, fructose and sucrose were run at different concentrations to create a calibration curve. Calibration curve for the glucose was considered for the detection of glucose concentration before and after the oxidation using sodium hypochlorite. There is a 90% decrease in glucose concentration when compared between R2 and P1. In contrast to the hypothesis of degradation of sugar molecules when oxidized using oxidants such as NaClO, can be evidenced due to a significant difference in concentrations between R2

and P1 (Fig 3). In R2, peak 9 and 10 represents glucose and xylose. In the analysis of samples P2 and R2, glucose exhibits a retention time (tR) of 14.9 minutes. In sample P1, the area percentage of glucose is 29.4%, indicating a substantial presence of this sugar. However, this percentage decreases significantly to 10.6% in sample P1, suggesting a notable reduction in glucose concentration. Similarly, xylose, having a retention time of 15.9 min for R2 and P1, area % decreased from 57.58 to 4.0. The decrease in area can be attributed to a decrease in the concentration of analyzed sugars present in both R2 and P1.

Based on the template, R2 was found to contain higher levels of glucose and xylose compared to other saccharides. Glucose and xylose are bonded together by glyosidic linkages, which facilitate the detection of these sugars. In contrast, P1 had lower glucose content along with other minor sugar molecules such as cellobiose, xylose, and arabinose. The intensity of all the sugars in P1 was significantly lower compared to R2, confirming the chemical changes to the sugar molecules. However, a notably high-intensity peak was observed at 10.8 min, indicating the presence of an unknown chemical that is likely an organic acid produced after oxidation (Fig 3). Even though it was not analyzed against any known standards.

14. Chemical structures of the reagents and sugars:

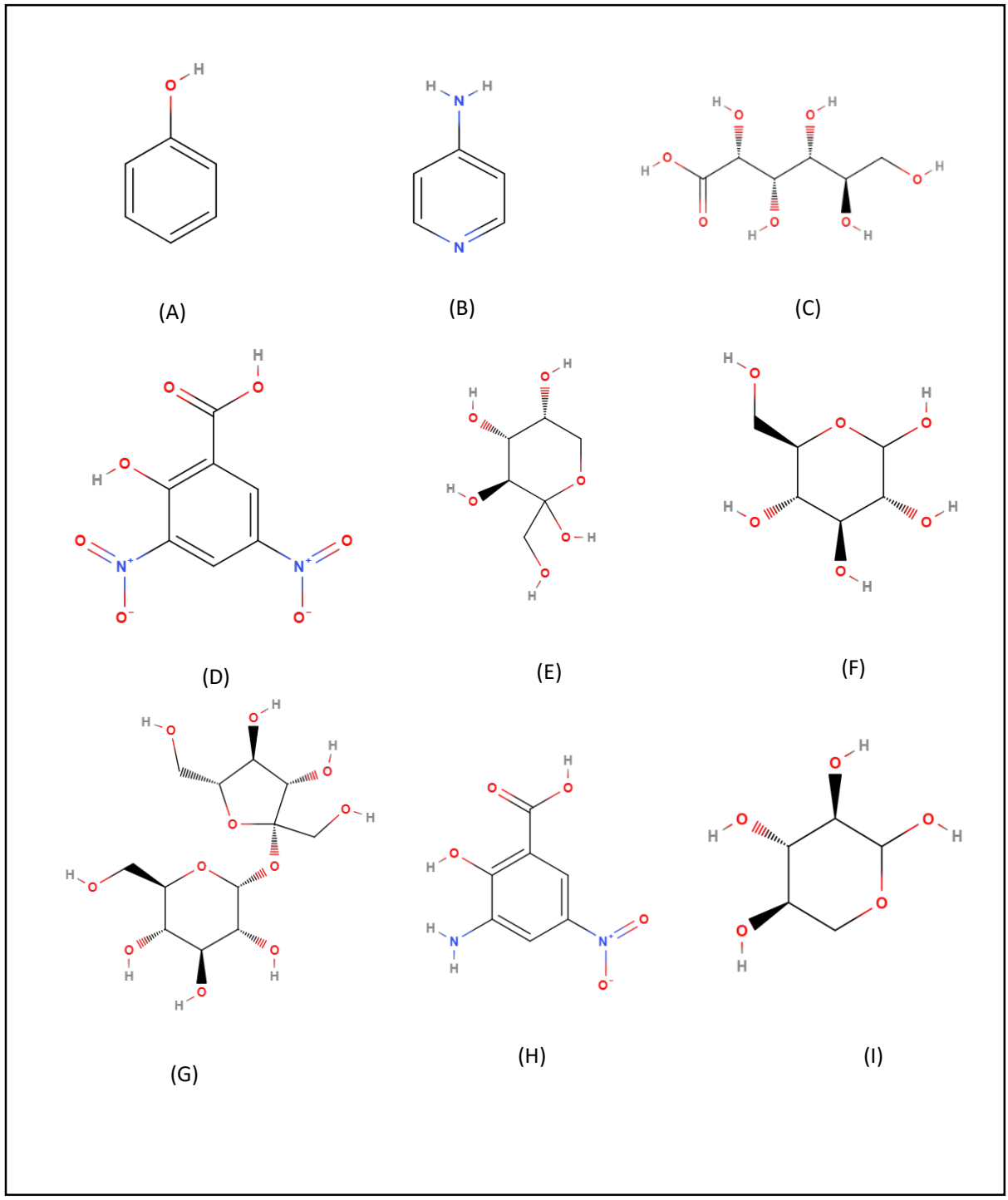


Figure 15

Structure	Name
A	Phenol
B	4-aminopyridine
C	Gluconic acid
D	3,5 dinitrosalicylic acid
E	Fructose
F	Glucose
G	Sucrose
H	3- Amino 5- nitrosalicylic acid
I	Xylose

15. SEM images:

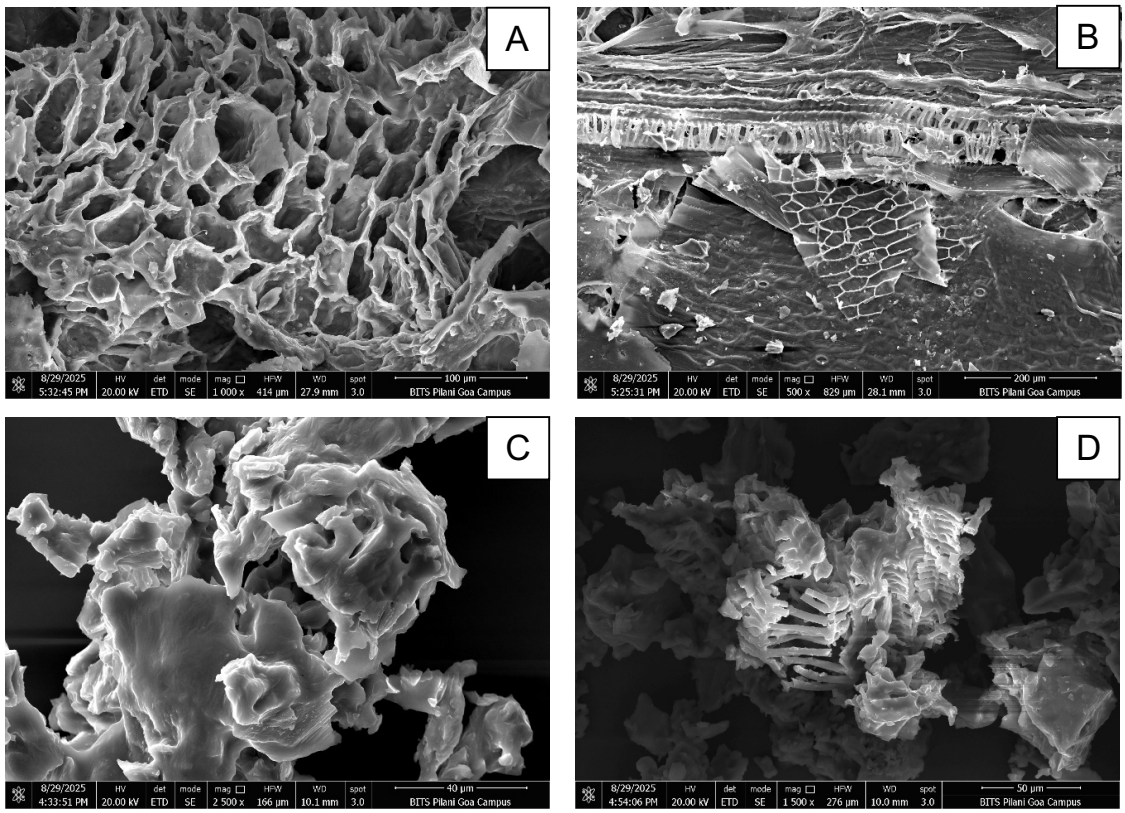


Figure 16

The SEM images of samples A and B in Figure 16 depict the raw onion sample dried at 70°C, illustrating the collapse of cell walls as moisture evaporates from the cell matrix. Samples C and D demonstrate the overlapping structure and the decomposition of sugars and pectin following oxidation.

16. Process snippets:

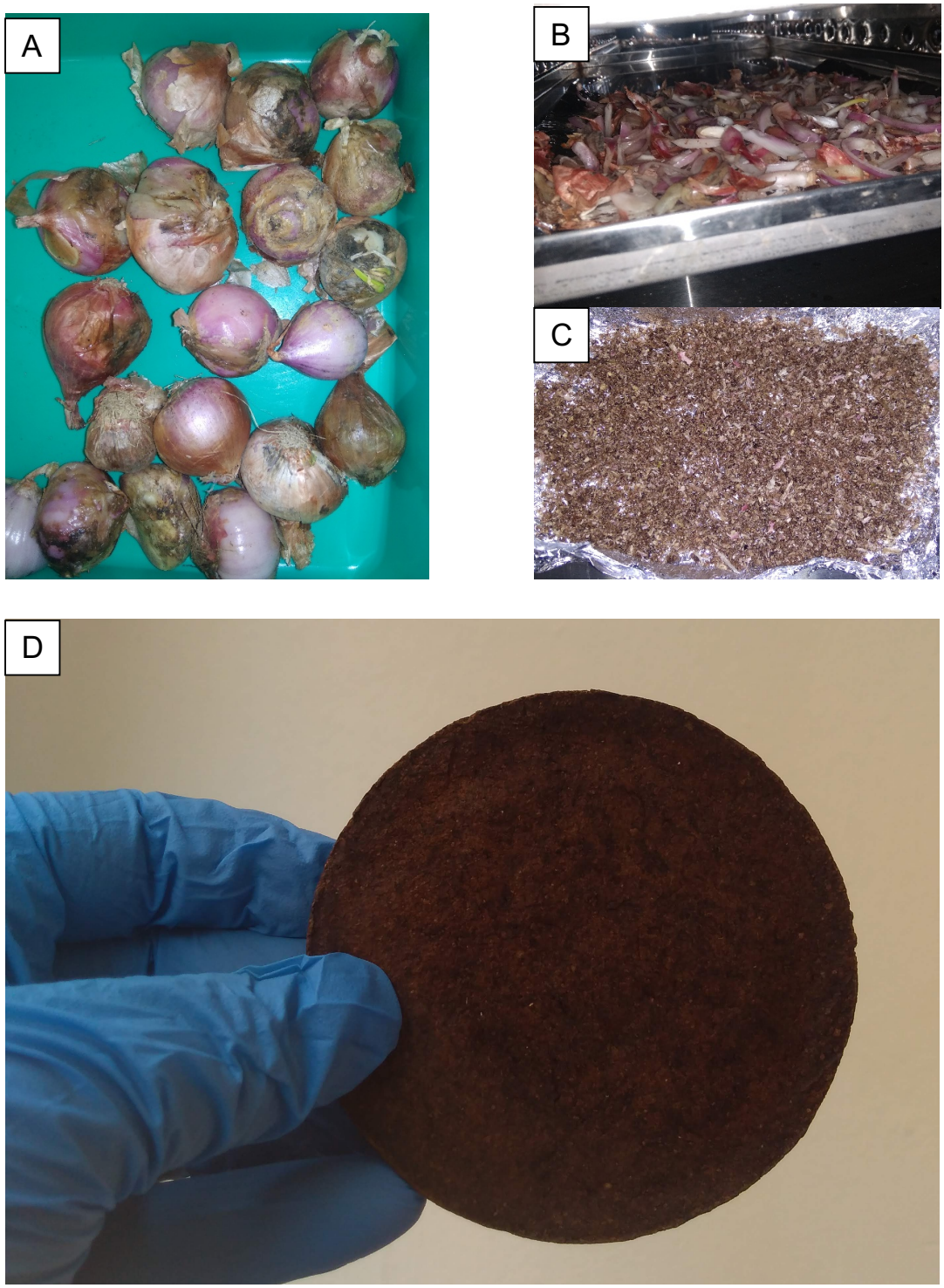


Figure 17

In the process snippets, figure 17A represents the waste onion along with roots, stems and peels which were used during the development of the process. Figure 17B represents the drying of the chopped onion samples in the tray dryer for moisture evaporation, and Figure 17C represents the chopped dried samples, which are finely chopped later to form onion powder R2 used in the experiment. Figure 17D represents the casted P2 sample.

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