Supplementary

Table S1: Central composite design matrix for Free Fatty Acidity.

Experiments	*Levels*					
	Oil (g)	Solvent (mL)	Indicator (mL)	KOH (N)	***Experimental Acidity***	***Predicted Acidity***
1	20	55	0.175	0.055	0.07	0.10
2	1	55	0.175	0.055	0.41	0.35
3	10.5	100	0.175	0.055	0.18	0.20
4	10.5	10	0.175	0.055	0.13	0.08
5	10.5	55	0.3	0.055	0.09	0.09
6	10.5	55	0.05	0.055	0.08	0.05
7	10.5	55	0.175	0.1	0.07	0.09
8	10.5	55	0.175	0.01	0.08	0.05
9	10.5	55	0.175	0.055	0.07	0.10
10	10.5	55	0.175	0.055	0.08	0.10
11	20	100	0.3	0.1	0.07	0.07
12	1	100	0.3	0.1	0.57	0.54
13	20	10	0.3	0.1	0.09	0.07
14	20	100	0.05	0.1	0.14	0.16
15	20	100	0.3	0.01	0.09	0.07
16	1	10	0.3	0.1	0.24	0.27
17	1	100	0.05	0.1	0.54	0.52
18	1	100	0.3	0.01	0.45	0.48
19	20	10	0.05	0.1	0.13	0.10
20	20	10	0.3	0.01	0.13	0.15
21	20	100	0.05	0.01	0.10	0.06
22	1	10	0.05	0.1	0.18	0.20
23	1	10	0.3	0.01	0.32	0.30
24	1	100	0.05	0.01	0.33	0.36
25	20	10	0.05	0.01	0.06	0.09
26	1	10	0.05	0.01	0.13	0.13
27	10.5	55	0.175	0.055	0.08	0.10

Table S2: Central composite design matrix for Peroxides Number.

Experiments	*Levels*					
	Oil (g)	Solvent (mL)	KI (mL)	Na_2SO_4 (N)	***Experimental P.N.***	***Predicted P.N.***
1	2.55	15	0.3	0.001375	11.21	10.68
2	0.1	5	0.5	0.00025	15.05	14.96
3	2.55	15	0.1	0.001375	11.08	11.57
4	2.55	15	0.3	0.001375	11.21	10.68
5	5	25	0.1	0.00025	9.60	9.75
6	2.55	25	0.3	0.001375	10.46	10.67
7	2.55	15	0.3	0.00025	10.47	9.69
8	5	15	0.3	0.001375	9.77	9.24
9	5	5	0.5	0.00025	4.52	4.85
10	2.55	15	0.3	0.001375	11.21	10.68
11	5	5	0.5	0.0025	7.74	7.85
12	5	25	0.5	0.00025	9.59	9.95
13	0.1	5	0.1	0.0025	15.18	14.80
14	0.1	25	0.5	0.0005	14.46	14.35
15	2.55	15	0.5	0.001375	10.82	10.76
16	5	5	0.1	0.0025	7.42	7.46
17	5	25	0.5	0.0025	10.51	10.61
18	0.1	25	0.1	0.0025	18.45	18.06
19	0.1	25	0.5	0.0025	18.97	18.65
20	0.1	15	0.3	0.001375	15.47	17.42
21	5	5	0.1	0.00025	7.68	7.96
22	2.55	5	0.3	0.001375	8.21	8.42
23	0.1	5	0.5	0.0025	17.19	16.87
24	2.55	15	0.3	0.0025	9.80	10.47
25	0.1	5	0.1	0.0005	17.31	17.13
26	2.55	15	0.3	0.001375	10.59	10.87
27	0.1	25	0.1	0.00025	19.63	19.47

Table S3: Central composite design matrix for Anisidine Number.

Experiments	*Levels*					
	Oil (g)	A solution (mL)	B solution (mL)	Anisidine (mL)	***Experimental A.N.***	***Predicted A.N.***
1	4	25	2.5	0.5	1.85	1.02
2	4	25	5	0.5	3.65	3.56
3	2.1	17.5	3.75	0.75	1.89	2.23
4	0.2	17.5	3.75	0.75	0.52	0.49
5	0.2	10	5	1	1.64	1.98
6	0.2	25	2.5	1	3.70	3.72
7	0.2	25	5	0.5	13.38	11.31
8	4	25	2.5	1	2.23	2.74
9	0.2	25	2.5	0.5	4.36	4.58
10	4	10	5	0.5	4.64	5.50
11	4	17.5	3.75	0.75	1.86	0.80
12	2.1	17.5	2.5	0.75	1.45	2.09
13	2.1	17.5	3.75	0.75	2.05	2.23
14	4	25	5	1	3.93	4.39
15	0.2	10	5	0.5	6.66	7.10
16	2.1	17.5	3.75	0.75	2.65	2.23
17	2.1	17.5	5	0.75	4.77	4.77
18	4	10	5	1	3.23	2.96
19	2.1	17.5	3.75	1	3.40	3.08
20	2.1	25	3.75	0.75	4.78	4.59
21	0.2	25	5	1	10.14	9.56
22	0.2	10	2.5	0.5	3.66	3.39
23	4	10	2.5	0.5	5.44	5.97
24	0.2	10	2.5	1	0.36	0.84
25	4	10	2.5	1	4.38	4.33
26	2.1	17.5	3.75	0.5	3.82	4.78
27	2.1	10	3.75	0.75	3.44	3.27

Table S4. Precision and accuracy of the miniaturized methods developed.

	Sample	Standard Method	Miniaturized method (intra-day)	Miniaturized method (extra-day)
Free Fatty Acidity (% of oleic acid)	1	0.073 ± 0.002	0.075 ± 0.002	0.076 ± 0.002
	2	0.076 ± 0.003	0.074 ± 0.001	0.075 ± 0.002
	3	0.070 ± 0.002	0.072 ± 0.003	0.073 ± 0.001
	4	0.076 ± 0.002	0.078 ± 0.002	0.077 ± 0.002
	5	0.071 ± 0.003	0.072 ± 0.002	0.073 ± 0.003
	6	0.077 ± 0.002	0.079 ± 0.003	0.078 ± 0.002
	7	0.073 ± 0.001	0.072 ± 0.002	0.073 ± 0.001
	8	0.079± 0.002	0.077 ± 0.002	0.078 ± 0.003
	9	0.077 ± 0.001	0.076 ± 0.003	0.075 ± 0.002
	10	0.073 ± 0.003	0.071 ± 0.002	0.072 ± 0.001
Peroxides Number (mgO_2/Kg)	1	11.01 ± 0.75	11.09 ± 0.95	11.08 ± 0.81
	2	11.11 ± 0.97	11.86 ± 0.77	11.75 ± 0.82
	3	11.40 ± 0.76	10.98 ± 0.86	11.06 ± 0.94
	4	11.52 ± 0.82	11.47 ± 0.94	11.42 ± 0.83
	5	10.95 ± 0.87	10.64 ± 0.86	11.20 ± 0.78
	6	10.18 ± 0.92	10.56 ± 0.72	10.81 ± 0.93
	7	10.37 ± 0.74	10.87 ± 0.77	11.04 ± 0.81
	8	11.79 ± 0.91	11.56 ± 0.86	11.76 ± 0.76
	9	11.68 ± 0.96	11.42 ± 0.97	11.56 ± 0.85
	10	10.10 ± 0.77	10.80 ± 0.88	10.45 ± 0.92
Anisidine	1	5.20 ± 0.20	4.35 ± 0.25	4.29 ± 0.22
	2	3.30 ± 0.22	3.15 ± 0.24	3.27 ± 0.23

Number	3	4.26 ± 0.20	4.41 ± 0.22	4.35 ± 0.22
	4	4.20 ± 0.26	4.16 ± 0.20	4.19 ± 0.24
	5	4.65 ± 0.24	4.72 ± 0.23	4.75 ± 0.21
	6	4.81 ± 0.21	4.93 ± 0.21	4.90 ± 0.20
	7	3.17 ± 0.20	3.15 ± 0.24	3.22 ± 0.23
	8	2.89 ± 0.21	2.97 ± 0.23	2.99 ± 0.24
	9	4.93 ± 0.26	5.07 ± 0.21	4.99 ± 0.26
	10	3.85 ± 0.23	3.71 ± 0.20	3.79 ± 0.22

Figure S1: Response surfaces for the interaction weight of sample – B solution volume for A.N. method.

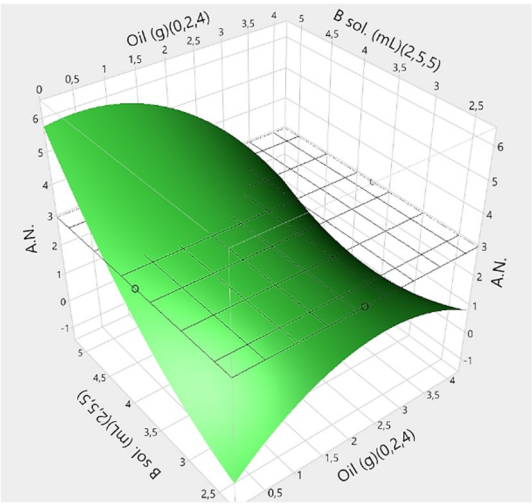

Figure S2: Response surfaces for the interaction A solution volume – B solution volume for A.N. method

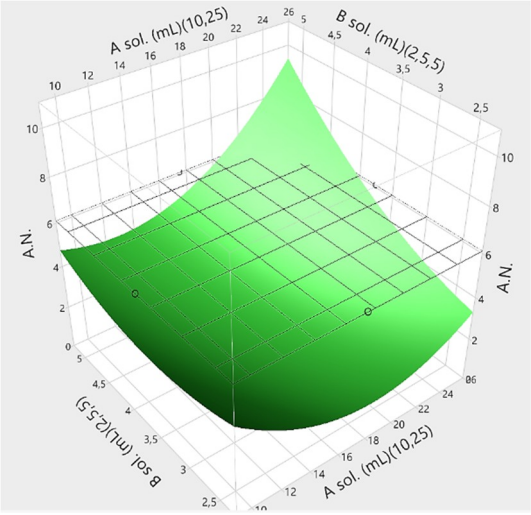
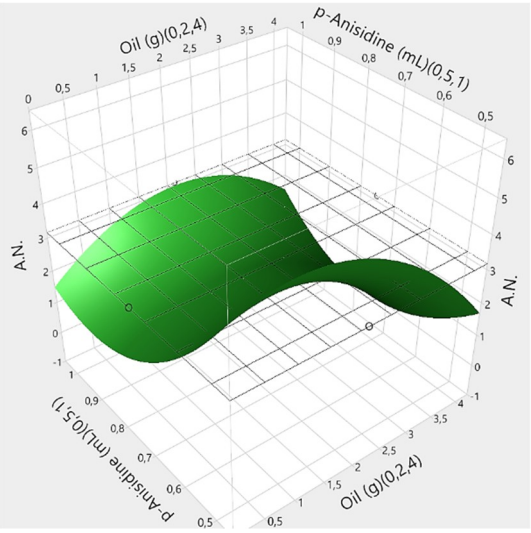
Figure S3: Response surfaces for the interaction weight of sample – Anisidine volume for A.N. method.