

Supplementary Information

Determination of Na, K, Mg and Ca in Biodiesel by LS F AAS and HR-CS F AAS: Studies that Supported the Proposal of the ABNT NBR 15556 Norm

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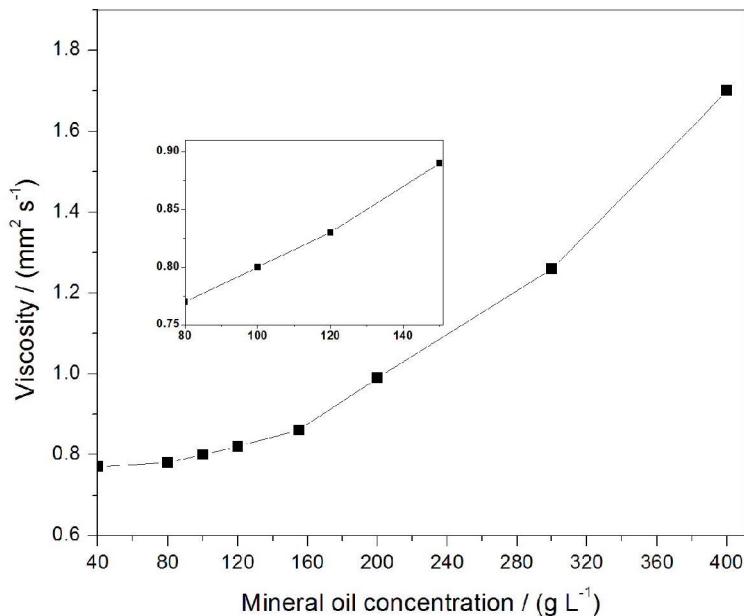


Figure S1. Viscosity of mineral oil in xylene solutions as function of the mineral oil concentration.

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#In memoriam

Dedicator: This work is dedicated to Professor Reinaldo Calixto de Campos who passed away on February 7th, 2012. The same day Reinaldo and some authors discussed the last details to finish this work. We would like to thank Reinaldo for his contribution to the development of the Analytical Chemistry in Brazil.

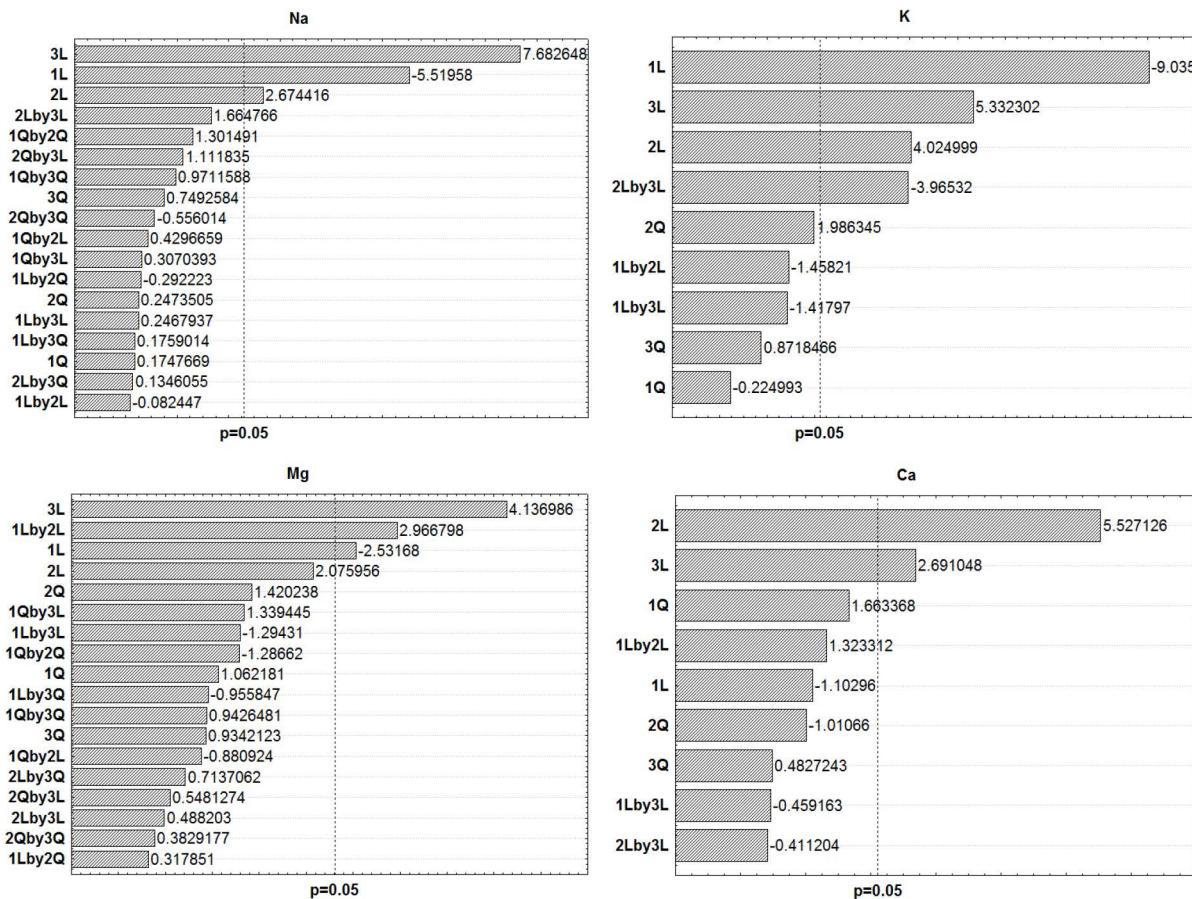


Figure S2. Pareto charts relatively to the factorial planning of the determination of Na, K, Mg and Ca in biodiesel by F AAS, (1) burner height, (2) acetylene flow rate and (3) aspiration rate.

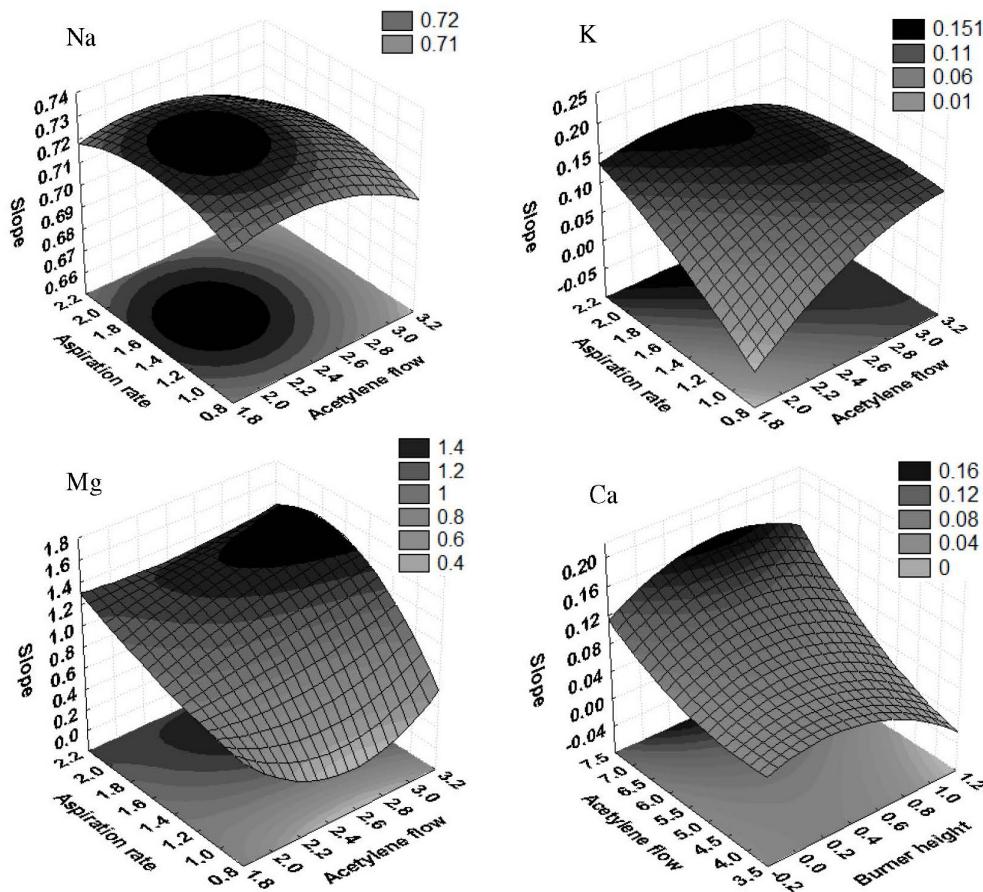


Figure S3. Response surfaces of the optimization experiment in the determination of Na, K, Mg and Ca in biodiesel by F AAS, slope ($L \text{ mg}^{-1}$), acetylene flow rate ($L \text{ min}^{-1}$), burner height (cm) and aspiration rate (mL min^{-1}).

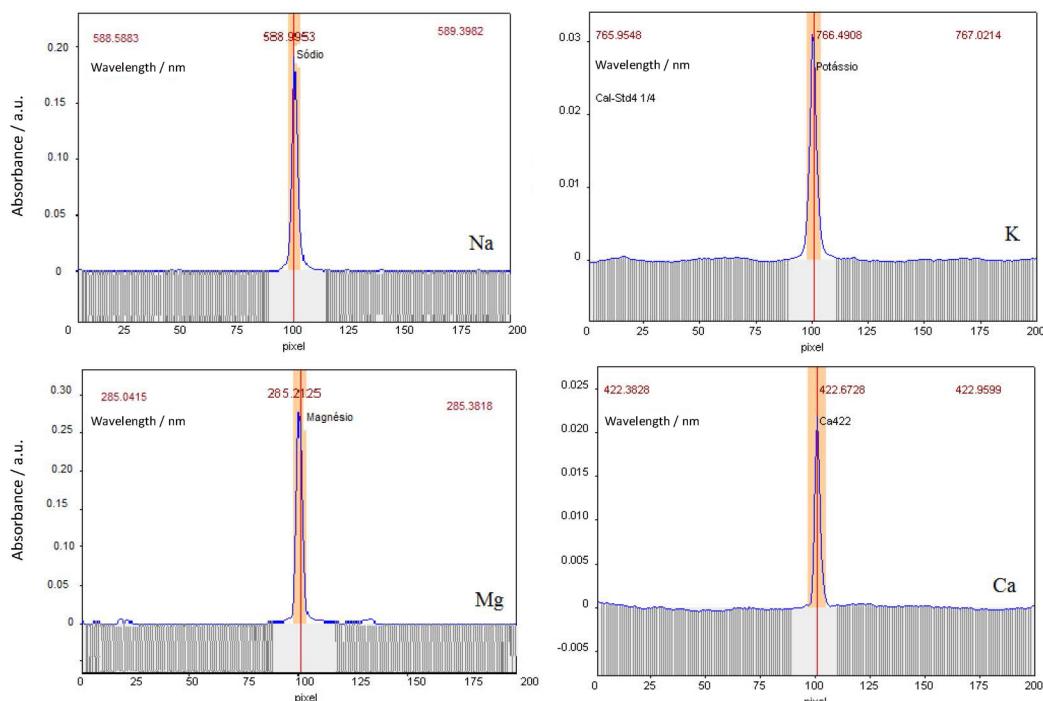


Figure S4. Spectral absorption profiles of Na, K, Mg and Ca in a biodiesel sample by HR-CS F AAS.

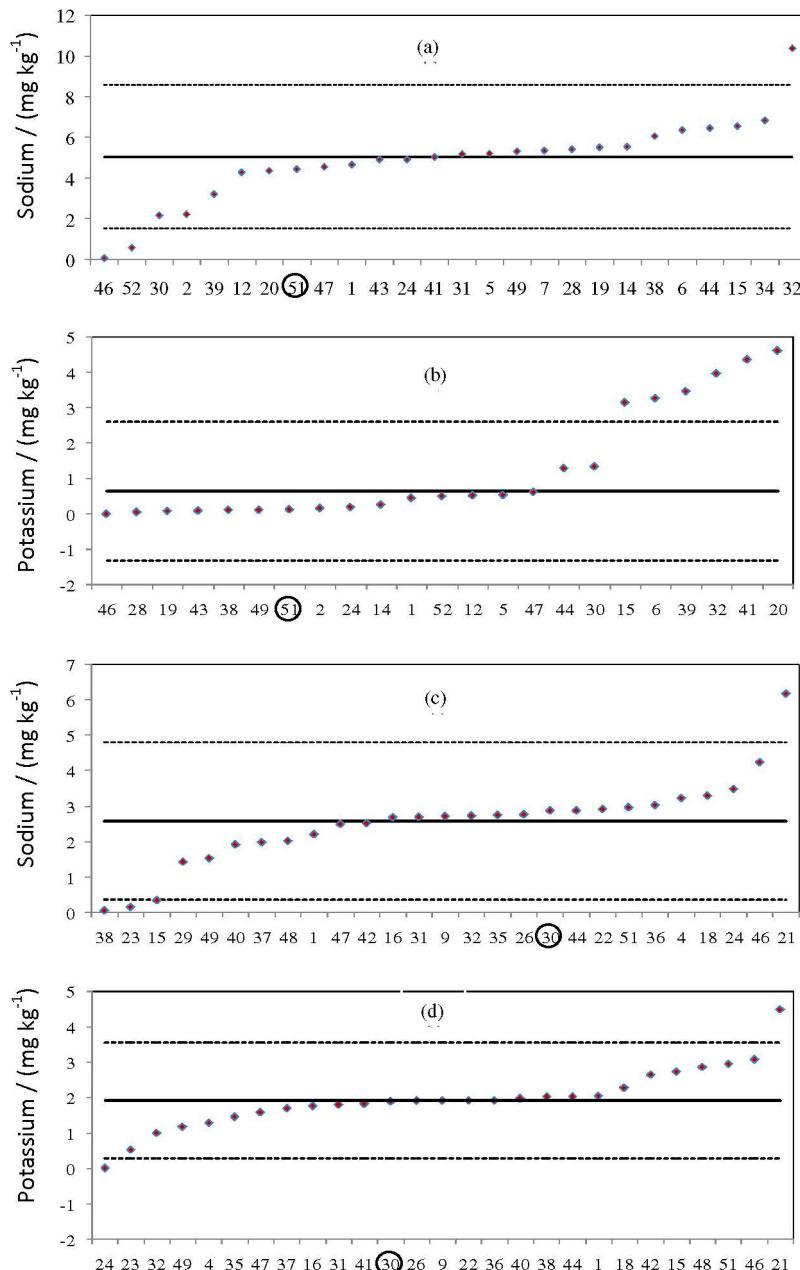


Figure S5. Graphical representation of the results of the 6th and 7th ANP interlaboratory exercises (PI), highlighting the results found in the present work using the ABNT NBR 15556 norm (51 and 30) in relation to the other participants: (a) Na 6th PI, (b) K 6th PI, (c) Na 7th PI and (d) K 7th PI, (◆) results, (—) average and (----) $\pm 3 \text{ s}$.

Table S1. Instrumental parameters for the determination of Na, K, Mg and Ca by LS F AAS and HR-CS F AAS

Parameters	LS F AAS				HR-CS F AAS			
	Na	K	Mg	Ca	Na	K	Mg	Ca
Lamp current / mA	10	10	4	4	hot spot mode			
λ / nm	589	766.5	285.2	422.5	588.99	766.49	285.21	422.67
Spectral resolution / nm - Number of pixels	0.8	0.8	1.2	1.2	7	7	7	9
Integration time / s	3.5	3.5	3.5	3.5	3.0	3.0	3.0	3.0
Aspiration rate / (mL min ⁻¹)	1.5	1.5	1.5	1.5	1.6	1.6	1.6	1.6
C ₂ H ₂ flow rate / (L min ⁻¹)	2.2	2.2	2.2	6.5	0.7	0.7	0.7	4.3
N ₂ O flow rate / (L min ⁻¹)	–	–	–	7.0	–	–	–	10.2
Air flow rate / (L min ⁻¹)	11.5	11.5	11.5	–	12.8	12.8	12.8	–
Observation height / cm	0.5	0.4	0.6	0.6	0.5	0.4	0.6	0.4

Table S2. Factors and their respective levels in the 3³ factorial planning

Factors	Levels					
	Na, K, Mg			Ca		
	-1	0	+1	-1	0	+1
1- Burner height / cm	0	0.5	1	0	0.5	1
2- C ₂ H ₂ flow rate / (L min ⁻¹)	2	2.5	3	4	5.5	7
3- Aspiration rate / (mL min ⁻¹)	1	1.5	2	1	1.5	2

Table S3. Response obtained from 3³ factorial planning for Na, K and Mg

Experiment	Burner height / cm	Acetylene flow / (L min ⁻¹)	Aspiration rate / (mL min ⁻¹)	Slope / (L mg ⁻¹)		
				Na	K	Mg
1	0	2	1	0.684	0.056	0.672
2	0	2	1.5	0.697	0.067	1.256
3	0	2	2	0.699	0.186	1.545
4	0	2.5	1	0.708	0.113	1.078
5	0	2.5	1.5	0.717	0.204	1.332
6	0	2.5	2	0.712	0.193	1.499
7	0	3	1	0.713	0.163	0.748
8	0	3	1.5	0.723	0.171	1.217
9	0	3	2	0.719	0.156	1.172
10	0.5	2	1	0.725	0.020	0.634
11	0.5	2	1.5	0.726	0.098	1.258
12	0.5	2	2	0.721	0.128	1.382
13	0.5	2.5	1	0.711	0.071	0.576
14	0.5	2.5	2	0.723	0.110	1.424
15	0.5	3	1	0.711	0.105	0.852
16	0.5	3	1.5	0.714	0.112	1.295
17	0.5	3	2	0.729	0.126	1.425
18	0.5	2	1	0.726	0.010	0.790
19	1	2	1.5	0.734	0.033	0.083
20	1	2	2	0.727	0.079	0.302
21	1	2.5	1	0.704	0.046	0.766
22	1	2.5	1.5	0.715	0.075	1.131
23	1	2.5	2	0.711	0.071	1.264
24	1	3	1	0.704	0.063	0.846
25	1	3	1.5	0.671	0.065	1.194
26	1	3	2	0.659	0.070	1.413
27	1	2.5	1.5	0.737	0.124	1.300

Table S4. Response obtained from 3³ factorial planning for Ca

Experiment	Burner height / cm	Acetylene flow / (L min ⁻¹)	Aspiration rate / (mL min ⁻¹)	Slope / (L mg ⁻¹) Ca
1	0	4	1	0.018
2	0	4	1.5	0.043
3	0	4	2	0.077
4	0	5.5	1	0.035
5	0	5.5	1.5	0.098
6	0	5.5	2	0.140
7	0	7	1	0.064
8	0	7	1.5	0.126
9	0	7	2	0.109
10	0.5	4	1	0.059
11	0.5	4	1.5	0.050
12	0.5	4	2	0.058
13	0.5	5.5	1	0.035
14	0.5	5.5	2	0.109
15	0.5	7	1	0.192
16	0.5	7	1.5	0.142
17	0.5	7	2	0.123
18	1	4	1	— ^a
19	1	4	1.5	— ^a
20	1	4	2	0.055
21	1	5.5	1	0.034
22	1	5.5	1.5	0.059
23	1	5.5	2	0.045
24	1	7	1	0.060
25	1	7	1.5	0.156
26	1	7	2	0.150
27	0.5	5.5	1.5	0.065

^aNo response.