Detection of Azo Dyes Using Carbon Dots from Olive Mill Wastes

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Supplementary Materials

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Procedure for the synthesis of WP-CDs [1]

The wet pomace, collected from an industrial olive extraction unit (olive mill), was dried at 105 °C until constant weight (~48 h) and then extracted with *n*-hexane in a Soxhlet apparatus during 4 h. After drying, the extracted pomace (EP; 8.0 g), dispersed in water (50 mL), and ethylenediamine (EDA; 712 µL; EDA/EP mass ratio = 0.08) were fed into an inox vessel of a high-pressure reactor (Parr model 4560, Parr Instruments Company, Moline, Ill, USA), deaerated by nitrogen flushing and stirred at 250 rpm in a nitrogen atmosphere. The contents of the reactor were heated with a temperature gradient of 15 °C/min until 250 °C and kept at this temperature for 72h. The reaction mixture was cooled to ca. 25 °C and filtered through a 0.20 µm cellulose membrane under vacuum. The filtrate was extracted five times with CH_2Cl_2 (25 mL). A brown aqueous solution of WP-CDs (ca. 16 mg/mL) was then obtained after the smooth removal of the residual organic solvent. The mass yield of WP-CDs was typically around 10%.

Structural and morphological characterization of WP-CDs

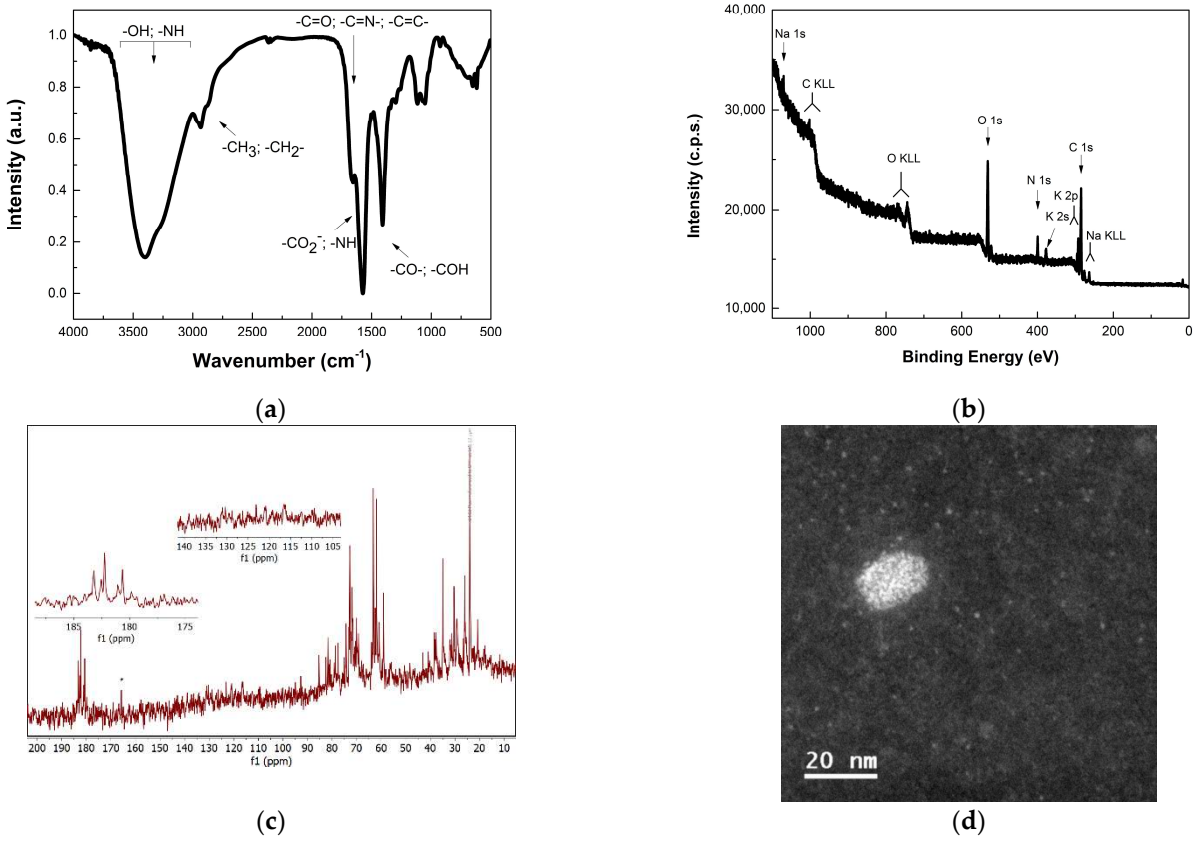
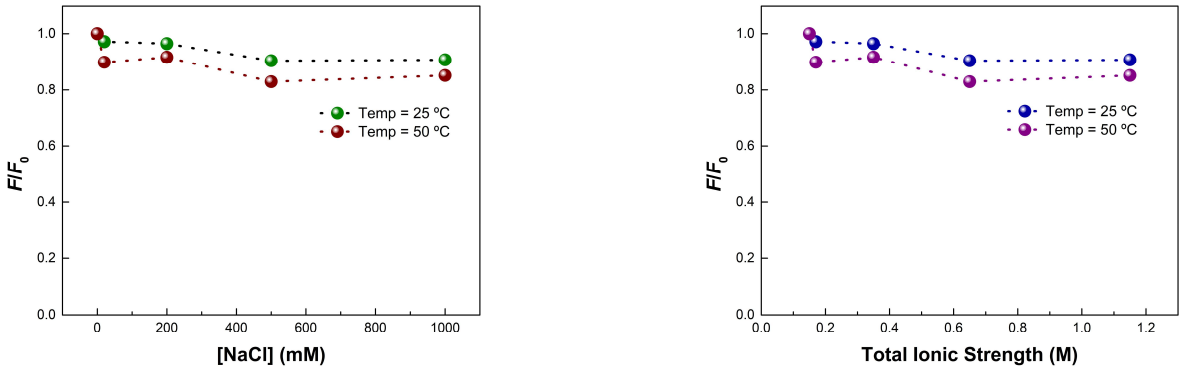


Figure S1. FTIR (**a**), wide XPS (**b**) ^{13}C NMR (**c**) spectra, and STEM micrograph (**d**) of WP-CDs. Full characterization details may be found in [1].

Effect of ionic strength on photoluminescence



(**a**) (**b**)

Figure S2. Effect of added NaCl (**a**) and total ionic strength (**b**) on the photoluminescence of WP-CDs (0.1 mg/mL) in phosphate buffer (50 mM, pH = 7.2) at 25 ºC and 50 ºC. Emission monitored at 426 nm upon excitation at 340 nm. Dotted lines drawn as an eye guide.

Detection of methyl orange (MO) in tap water

Table S1. Detection of MO in tap water.[1]

Sample	[MO] spiked (μM)	[MO] found (μM)	Recovery (%)	RSD (%; n = 3)
1	0	Not detected	-	-
2	10	10.38 ± 0.17	103.8 ± 1.7	1.63
3	15	14.12 ± 0.11	94.1 ± 0.7	0.76
4	20	20.77 ± 0.02	103.8 ± 0.1	0.09

[1] WP-CDs probe was used at a concentration of 0.1 mg/mL; tap water from the Lisbon public water system (EPAL) [2]. Emission monitored at 426 nm upon excitation at 340 nm.

References

1. Sousa, D.A.; Ferreira, L.F.V.; Fedorov, A.A.; Rego, A.M.B.; Ferraria, A.M.; Cruz, A.B.; Berberan-Santos, M.N.; Prata, J.V. Luminescent Carbon Dots from Wet Olive Pomace: structural insights, photophysical properties and cytotoxicity. *Molecules* **2022**, *27*, 6768. https://doi.org/10.3390/molecules27196768.
2. Dados da Qualidade da Água/Torneiras da Cidade de Lisboa. EPAL, Grupo Águas de Portugal, 2022. https://www.epal.pt/EPAL/menu/%C3%A1gua/divulga%C3%A7%C3%A3o-de-dados-da-qualidade-da-%C3%A1gua/controlo-legal/torneiras-na-cidade-de-lisboa (accessed on 3 November 2022).