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ORIGINAL ARTICLE

A chemometric study: Automated flow injection analysis method for the quantitative determination of humic acid in Ilgın lignite

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Abstract A rapid, sensitive and provident flow injection analysis (FIA) method was developed within the framework of a chemometric approach for the quantification of humic acid (HA) in the lignite obtained from Ilgın, Konya, Turkey. The proposed method allows automatic determination of 60 samples per hour over a wide calibration range (0–2000 mg L^{-1}, R^2: 0.9988) and needs only 10 μL of sample at a flow rate of mobile phase (X_1), 2 mL min^{-1}; pH of mobile phase (X_2), 8, and system temperature (X_3), 20 °C. The limits of detection (LOD) and quantification (LOQ) were calculated as 9.18 mg L^{-1} and 30.60 mg L^{-1}, respectively, and the relative standard deviation (RSD) for 500 mg L^{-1} HA was calculated as 3.44 (n: 9). It was revealed that the standard deviation (SD) values of the proposed FIA method are lower than those of the spectrophotometric method.

1. Introduction

Turkey has substantial reserves of lignite; because of this, the Turkish energy system is based on lignite exploitation. However, high ash and high sulfur content limit the use of these coals for economical practice and in power generation cause serious environmental problems (Akgun et al., 1989; Karaca et al., 1997; Lin et al., 1997). For these reasons, great interest has grown in the possible alternative use of lignites as a source of soil amendments in order to maintain and increase the content of natural organic matter (NOM) of the soil (Schobert, 1995; Peuravuori et al., 2006).

Humic substances (HSs) are the most dominant fraction of NOM in the soil. HSs are a series of different molecular weight, light-brown to black-colored complexes and heterogeneous organic polymers formed by secondary synthesis reactions (Stevenson, 1982). These substances can be classified into three main fractions based on their solubilities in alkaline and acidic extraction solutions. Humic acid (HA) fraction is soluble in alkaline solutions but insoluble in acidic solutions; fulvic acid (FA) fraction is soluble in both alkaline and acidic solutions; while humin is insoluble in both solutions (Stevenson, 1982; Schnitzer, 1982). Among these, the

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predominant fraction is HA, which is very active in interacting with organic and inorganic chemicals as compared to other fractions (Kishi, 1988; Senesi, 1993). This substance plays an important role in soil conservation, for water-holding capacity, and for the complexation of metals in terrestrial and aquatic systems (Swift, 1996; Hayes and Malcolm, 2001; Hayes and Graham, 2000).

Lignite that is another soil type is usually used for the production of HA which is in the form of alkali-soluble humate salts (Peuravuori et al., 2006). The characteristics (size, chemical composition and functional groups) may differ considerably, depending on the origin and age of the material (Stevenson, 1982; Fong and Mohamed, 2007). Because of these properties, determination of HA in lignite has great

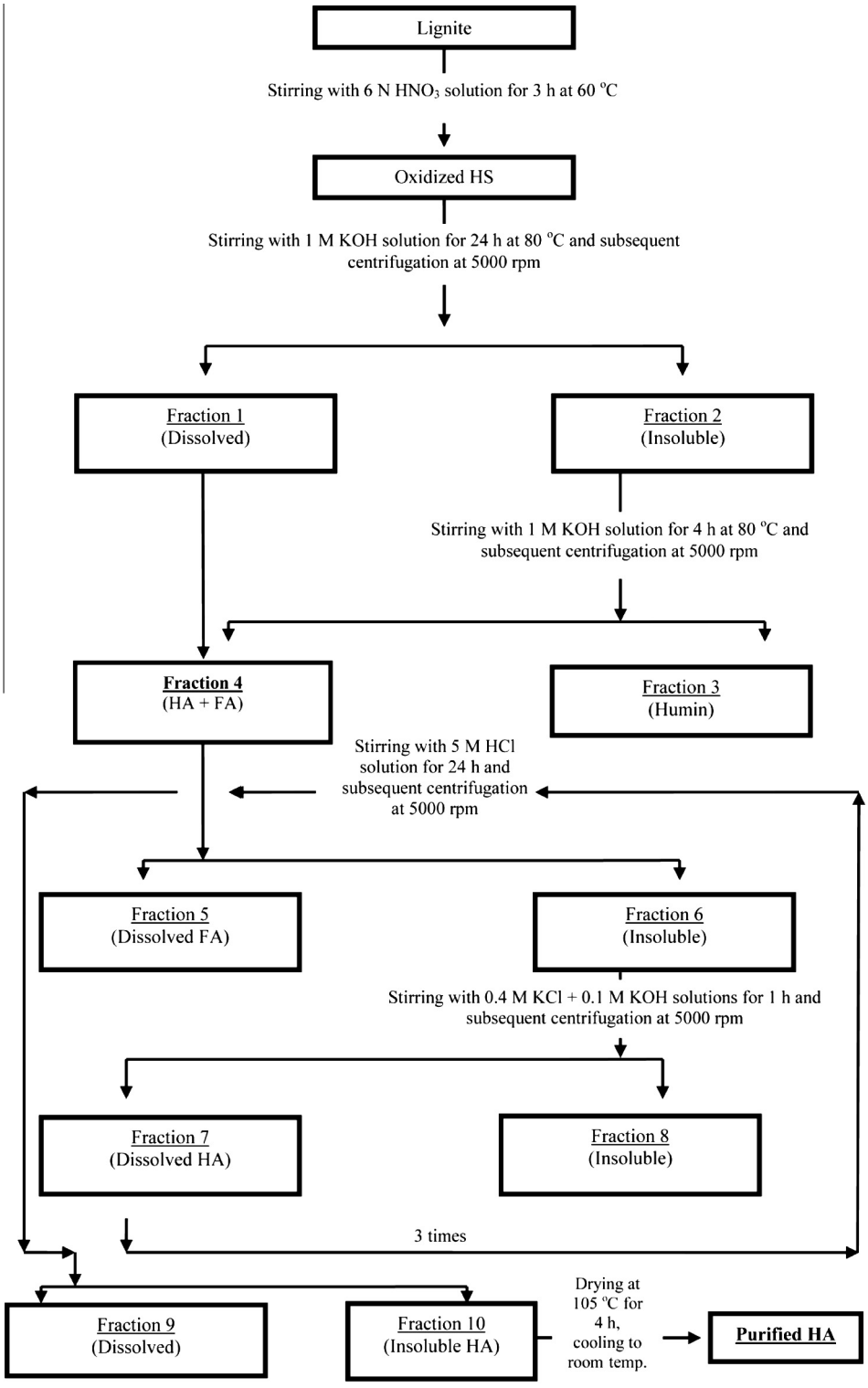

Figure 1 Extraction and purification procedure of lignite samples (Tarhan, 2011).