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Ultraviolet Combined with Microwave Radiation for Digestion of Commercial Animal Feed and Further P and S Determination by IC

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Introduction

Among minerals essential for the animal physiological system, special attention should be given to P and S, taking into account that these macronutrients are involved in functions such as reproduction, growth, energy metabolism, osmotic and anion-cation balance.[1] However, considering that commercial animal feed usually presents a high content of protein and fat, it is important to develop suitable sample preparation methods, which could interfere in the accuracy of results for P and S determination. The microwave-assisted ultraviolet digestion (MW-UV) can be an alternative presenting high efficiency of digestion (with low residual carbon content -RCC) even using diluted acids.[2,3] In this work was proposed the use of MW-UV for digestion of commercial animal feed and subsequent P and S determination by ion chromatography (IC).

Results and Discussion

Bovine, horse, rabbit, fish and chicken feed were purchased in a local market (Pelotas, RS, Brazil), grounded and dried (65 °C/12 h). One sample was arbitrarily selected for the optimization of methods. In MW-UV method, samples (500 mg) were digested using a UV lamp inside of digestion vessels, and 10 mL of HNO_3 solutions (2 to 10 mol L^{-1}) were evaluated for digestion. For comparison of results, conventional microwave-assisted wet digestion (MW-AD) using diluted HNO_3 solutions were also performed. Both digestion methods were performed using a microwave oven (Multiwave 3000^{TM,} Anton Paar), with the following program: *i)* 600 W/6 min; *ii)* 800 W/10 min; and *iii)* 0 W/20 min. Determination of P and S in digests was carried out using an ion chromatograph (IC 850, Metrohm, Switzerland), and C by inductively coupled optical emission spectrometry (ICP OES, Spectro Ciros CCD, Spectro Analytical Instruments, Germany). Additionally, methods for P (965.17) and S (980.02) determination in animal feed, recommended by the Association of Analytical Communities (AOAC), were also performed. In AOAC methods, samples were digested in an open system with conventional heating, and P and S were determined by spectrophotometric and gravimetric methods, respectively. The accuracy of methods was evaluated by recovery tests and by the analysis of a reference material (RM) of bovine muscle (NIST 8414). Results for RCC after digestion of animal feed (Fig. 1) shown high values of carbon in the final solutions when was used the AOAC methods. It probably occurs due to the reagents did not achieve enough temperature for digestion using an open system. On the other hand, when MW-AD method was used, lower RCCs (< 314 mg L^{-1}) were obtained by using 7 mol L^{-1} HNO_3 or higher concentration. However, the use of 2 mol L^{-1} HNO_3 by MW-UV method was enough to obtaining similar efficiency of digestion (RCC: 304 ± 21 mg L^{-1}) when compared to the conventional MW-AD method. Therefore, MW-UV proposed method allows the use of diluted acid without decreasing the efficiency of digestion.

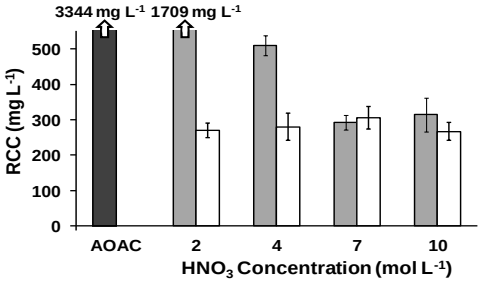

Figure 1. Determination of carbon in animal feed by ICP OES after digestion using AOAC ■,MW-AD ▨ and MW-UV □methods (n=3).

Moreover, using MW-UV proposed method (2 mol L^{-1} HNO_3), recoveries for P and S were around 98%, and agreements with the informed values in RM were better than 94% for both analytes. Additionally, the use of diluted acid was more suitable for IC analysis. The limits of detection for P and S, using optimized conditions, were 670 and 68 µg g^{-1}, respectively. In addition, it is important to mention that the results obtained by the AOAC method for P and S were not in agreement (< 60%) with those obtained by the proposed method, probably due the sample matrix has not been decomposed efficiently (RCC > 3344 ± 539 mg L^{-1}). Phosphorus and S were also determined in other animal feed using the proposed method and concentrations ranged from 28357 to 10026 µg g-1 for P and 4601 to 2259 µg g^{-1} for S.

Conclusions

The proposed method was suitable for digestion of commercial animal feed and determination of P and S by IC. Furthermore, using the proposed method lower blanks values and LOD were obtained, and the final solutions were suitable for IC analyses.

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