GRAVITATIONAL SEDOMENTOMETRICAL ANALYSES APPLIED TO THE WHEAT STRAW LIGNIN

ANALIZE SEDIMENTOMETRICE GRAVITAȚIONALE APLICATE LIGNINEI DIN PAIE DE GRÂU

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Abstract. *The lignin stands out by a very large range of applications in extremely various domains. The adsorption-desorption capacity, ion exchange capacity and its catalytic properties are just a few specific characteristics which are emphasizing the importance of harnessing the lignins. In this paper is shown by sedimentometrical analyses that lignin can be used inagriculture and zootech.*
Key words: *sedomentometrical analyses, wheat straw lignin, density, pycnometer, sedomentometrical curves.*

Rezumat. *Lignina se remarcă printr-o gamă foarte largă de aplicații în domenii extrem de diverse. Capacitatea de absorbție-desorbție, capacitatea de schimb ionic, proprietățile catalitice sunt doar câteva repere specifice care recomandă şi evidențiază importanța valorificării ligninelor. În această lucrare, se arată prin sedimentarea gravitațională, că lignina se poate utiliza în agricultură şi zootehnie, industria celulozei şi hârtiei, construcții, metalurgie sau cataliză. sedimentometrical analyses that lignin can be used inagriculture and zootech.*
Cuvinte cheie: *analize sedimentometrice, lignină din paie de grâu, densitate, picnometru, curbe sedimentometrice.*

INTRODUCTION

At a global level, lignin resulted from cellulose fabrication or technologies of hydrolysis of vegetal mass can be considered as raw material with high capitalization potential, because of its provenience from regenerating sources and due to reduced price. Lignin is a macromolecular compound, much more active than cellulose or other natural polymers, due to functional groups contained in its macromolecule, constituting the main aromatic component of vegetal tissues, standing for 20%-30% of the mass at superior plants, where it is present within the cellular membrane and in intercellular spaces (Ungureanu E., 2011; Hiementz et al., 1997). The structure of wheat straw lignin is naturally variable: macro-porous, porous or micro-porous. (Ungureanu et al, 2012; Odochian,1989).

Up to now, the attempts made at creating an industry which can capitalize lignin have registered only partial success.

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Fig. 1 - Wheat straw lignin (dust)

One of the pursued directions refers to ameliorating the properties of lignin resulted from processes of chemical wood and annual plants processing through reactions that may lead to increased functionality and diversification of the fields of use for lignin. Due to its regeneration, capacity through photosynthesis, vegetal biomass and its components (including lignin) will become in the future sources of raw material with a high degree of capitalization.

In this paper is shown by sedimentometrical analyses that lignin can be used in agriculture and zootechnics, cellulose and paper industry, constructions, metallurgy or as catalysts.

MATERIAL AND METHOD

The following materials have been used:

- Wheat straw lignin (L_1), offered by Granit Recherché Dévelopement, having the characteristics described in table 1

Table 1

Characteristics of wheat straw lignin L_1

Characteristics	**L_1**
Relative humidity, %	5.00
Ash, %	2.30
pH in suspension	2.70
Manganese, %	0.7
Nitrogen, %	1
Uronic acid, %	0
Solubility in acids, %	1
Insolubility in acids, %	90

- RS-71 Tensio-tixometer gravimetric sedimentation balance;
- Steel ball crusher;
- Toluene;
- Distilled water.

Methods:

- picnometer method;
- gravitational sedimentometrical method.

Work procedure***:*** 45 g of wheat straw lignin were weighed, crushed for 30 min. and dissolved in 1L of distilled water. The electrostatic forces of attraction between the hydroxylic groups of the lignin and the dipoles of the dissociated water are so powerful that a colloidal-hydric aggregate is formed and its volume is smaller that the sum of volumes that interact (water-lignin). Experimental data were statistically processed with the aid of the *Unscrambler* application.

RESULTS AND DISCUSSIONS

Based on the standard curve (fig.2) 10 sedimentation curves were plotted according to the dependence q(g) = f(t, s), and the experimental data are also listed in tables.

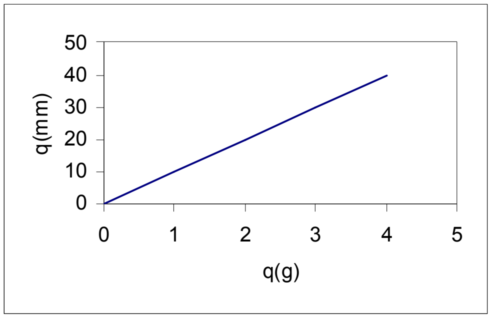


Fig. 2 - The standard curve

10 sedimentation curves in q(mm) = f(t, s) coordinates were obtained using RS-71 Tensio-tixometer under constant conditions (mass lignin = 45 g/L water).

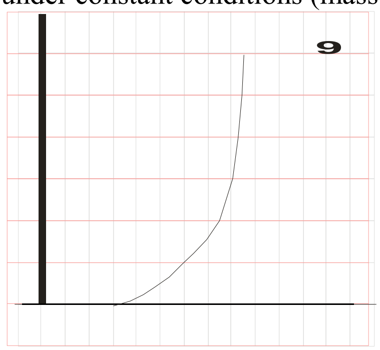


Fig. 3 - Exemple of sedimentation curve

These sedimentation curves were also listed in table 2.

Table 2

Parameter values of the sedimentation curves

No. det.	t (mm)	t (sec)	q (mm)	q (g)
1	3	7,85	3,50	0,25
2	6	15,7	5,0	0,40
3	9	23,49	7,0	0,59
4	12	31,40	8,0	0,60
5	15	39,25	9,0	0,73
6	18	47,10	10,5	0,80
7	21	54,95	11,0	0,86
8	24	62,80	11,5	0,89

9	27	70,65	12,0	0,90
10	30	78,50	12,5	0,99
11	33	86,35	12,5	0,99
12	36	94,20	13,0	1,01
13	39	102,05	13,5	1,04
14	42	109,90	13,5	1,04
15	45	117,75	14,0	1,11
16	48	125,60	14,0	1,11
17	51	133,45	14,5	1,17
18	54	141,30	14,5	1,17
19	57	149,15	14,5	1,17
20	60	157,00	14,5	1,17

Further on it was obtained the most expected sedimentation curve plotting the values of sediment quantity, q(g) and time t(s), for the reproductible measurements (2, 3, 4, 5, 9) (fig. 4).

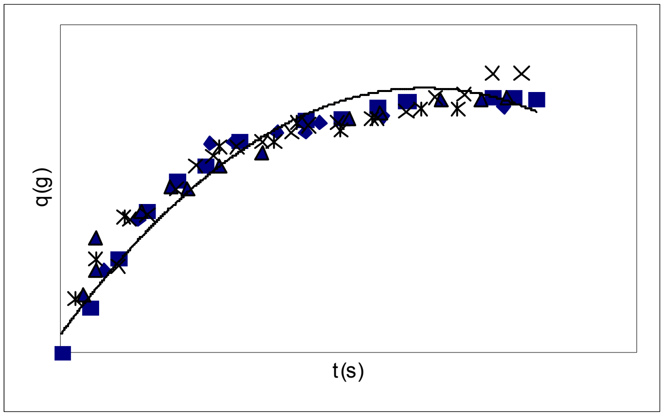
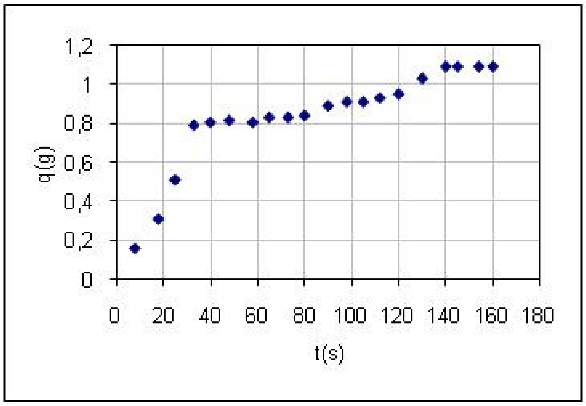


Fig. 4- General sedimentation curve

Measurements 1, 6, 7 and 10 are not reproductible due to a non-uniform distribution of the scattered particles obtained before the recordings (fig. 5a and b).



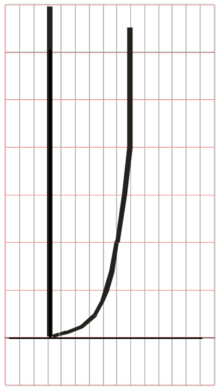


Fig. 5 - Sedimentation curves using:
a) q(g) = f(t, s) dependence b) tensio-tixometer q(mm) = f(t, min.)

dependence

Based on the general theory of sedimentation in gravimetric field of micro-heterogeneous systems, the radius boundaries of the scattered particles in ground lignin were evaluated. According to the determinations performed the amount of deposited lignin was Q = 1.18 g. In order to determine the density of lignin, the picnometer method was employed. In table 3 are presented the values obtained experimentally by weighing or theoretically determined.

Table 3

Density of lignin measured by picnometer method

m_1 (g)	m_2 (g)	m_3 (g)	m_4 (g)	ρ_s (g/cm^3)
13,6512	14,6515	23,0630	22,4048	2,5307
13,6512	14,6508	23,0622	22,4048	2,5282
13,6512	14,6486	23,0642	22,4048	2,5539

m_1 – empty pycnometer mass; m_2 – pycnometer mass + solid; m_3 – pycnometer mass + solid + liquid; m_4 – pycnometer mass + liquid (toluene); ρ_s - solid density

By graphical derivation of the sedimentation curve (Fig.3), the sedimentation rates were obtained at certain periods of time, t = 0, 14, 30, 70, 80, 90, 100, 105 s. These rates were used to determine various fractions radii of the disperse system (table 4).

Table 4

Sedimentation rates corresponding to the most expected sedimentation curve for the reproductible results

Time (s)	Sediment quantity (g)	Sedimentation rates (mm/s)
0	0	0,042
14	0,2	0,0164
30	0,6	0,0140
70	0,94	0,0035
80	0,97	0,0026
90	1,00	0,0017
100	1,03	0,0015
105	1,04	0,0150

Based on the resulted sedimentation rates, the particle radii of lignin were obtained (table 5).

Table 5

Values of disperse particle radii of lignin obtained by sedimentation in gravitational field.

Fractions number	Dimension of superior and inferior sieve mesh (mm)	Diameter a_i (mm)	Beam r_i (mm)	
			By rieving	By sedimentation gravitational feils
1	1,25 – 1,10	1,125	0,562	-
2	1,0 – 0,05	0,815	0,407	0,30 (0)
3	0,63 – 0,25	0,440	0,220	0,1570 (14)
4	0,25 – 0,20	0,225	0,1125	0,140 (30)
5	0,20 – 0,125	0,1625	0,0812	0,0740 (70)
6	0,16 – 0,10	0,130	0,0650	0,0630 (80)
7	0,10 – 0,09	0,095	0,0470	0,0520 (90)
8	0,09 – 0,08	0,085	0,0425	0,048 (100)
9	0,08 – 0,07	0,075	0,0370	0,040 (105)

CONCLUSIONS

1.The sedimentometrical method applied in gravitational field confirms that the reproducibility of the experimental data depends on the uniform distribution degree of the analyzed disperse particle.

2.In order to determine the lignin density, the picnometer method was success fully employed.

3.The variation range of the disperse particle radius in ground lignin can be determined either by sieving or by sedimentation in gravitational field.

4.The analysis of the values obtained for particle dimensions of wheat straw lignin using both methods leads to a general conclusion that the obtained data are reproducible.

5.Using the gravitational sedimentation, it can be determined in a shorter period of time the particle dimensions comparing with the sieving method, which is a much more complex one.

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