

GRAVITATIONAL SEDIMENTOMETRICAL ANALYSES APPLIED TO THE SARKANDA GRASS LIGNIN

ANALIZE SEDIMENTOMETRICE GRAVITAȚIONALE APLICATE LIGNINEI DIN IARBĂ

UNGUREANU Elena^{1*}, UNGUREANU O.C.², TROFIN Alina¹
ARITON Adina-Mirela³, JIȚĂREANU Carmenica Doina¹, POPA V.I.⁴

*Corresponding author e-mail: eungureanu@uaiasi.ro

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 in agriculture and zootech. The sarkanda grass lignin (L₂) offered by the Granit Recherche Developement S.A. company, Lausanne-Schwitzerland was synthesized from annual plants.*

Key words: sedimentometrical analyses, sarkanda grass lignin, density, pycnometer, sedimentometrical curves

Rezumat. *Lignina se remarcă printr-o gamă foarte largă de aplicații în domeniul extrem de diverse. Capacitatea de absorbție-desorbție, capacitatea de schimb ionic, proprietățile catalitice sunt doar câteva repere specifice care recomandă și evidențiază importanța valorificării ligninelor. În această lucrare, se arată prin sedimentarea gravitațională, că lignina se poate utiliza în agricultură și zootehnie, Lignina sarkanda grass lignin (L₂) oferită de firma Granit Recherche Developement S.A. Lausanne-Elveția a fost sintetizată din plante anuale.*

Cuvinte cheie: analize sedimentometrice, lignină din iarbă, densitate, picnometru, curbe sedimentometrice

INTRODUCTION

Lignin comprises as much as 30 percent of wood's mass and therefore the development of novel technologies for the use of lignin in composites is an attractive, environmentally intelligent goal (Area *et al*, 2014).

Until now, the attempts to develop an industry, which would put in good use residual lignin, had only partial success. This situation is due especially with a lot of difficulties met in usage and processing of the lignin (Popa, 2015, 2016).

These difficulties are connected to its complex chemical structure, different chemical units, chemical bonds with other components and a great capacity of this

¹University of Agricultural Sciences and Veterinary Medicine Iasi, Romania

²“V.Goldiș” West University of Arad, Romania

³Station for Cattle Breeding, Dancu, Iasi, Romania

⁴“Gh. Asachi” Technical University of Iasi, Romania.

natural aromatic polymer to be modified irreversibly during chemical reactions or under the action of physical agents. It is known that lignin has a very complex structure, which varies depending on the plant species, separation method and modification reactions that may induce particular characteristics (Ungureanu, 2011).

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 (Ungureanu *et al*, 2006, Căpraru *et al*, 2009).

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:

- Sarkanda grass lignin (L₂), offered by Granit Recherché Développement, having the characteristics described in table 1.

Table 1

Characteristics of sarkanda grass lignin L ₂	
Characteristics	L ₂
Relative humidity, %	5.20
Ash, %	2.40
pH in suspension	3.20
Manganese, %	0.69
Nitrogen, %	1.2
Uronic acid, %	0
Solubility in acids, %	2
Insolubility in acids, %	87

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

Methods

- picnometer method;
- gravitational sedimentometrical method.

Work procedure: 45 g of sarkanda grass 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 than 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.1) 10 sedimentation curves were plotted according to the dependence $q(g) = f(t, s)$, and the experimental data are also listed in tables.

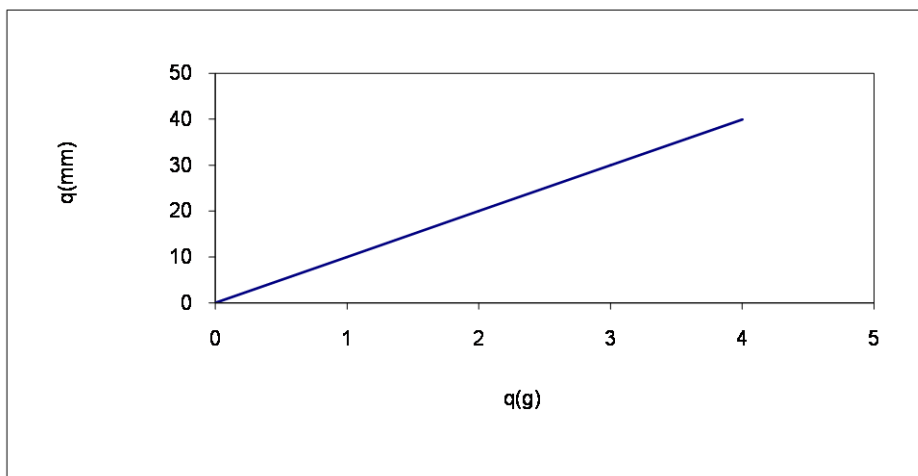


Fig. 1 The standard curve

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

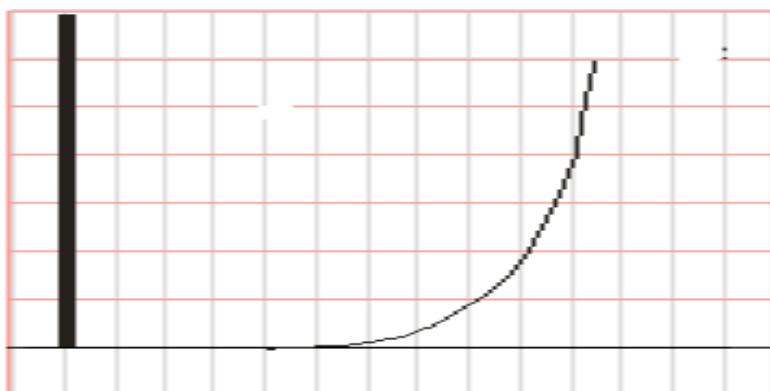


Fig. 2 Exemple of sedimentation curve

These sedimentation curves were also listed in table 2.

Parameter values of the sedimentation curves

No det.	t (mm)	t (sec)	q (mm)	q (g)
1	3	7.85	3.51	0.25
2	6	15.7	5.1	0.41
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	10.6	0.82
7	21	54.95	11.0	0.86
8	24	62.80	11.4	0.85
9	27	70.65	12.0	0.90
10	30	78.50	12.6	0.98
11	33	86.35	12.7	0.98
12	36	94.20	13.0	1.02
13	39	102.05	13.6	1.03
14	42	109.90	13.6	1.04
15	45	117.75	14.0	1.11
16	48	125.60	14.0	1.11
17	51	133.45	14.6	1.18
18	54	141.30	14.6	1.18
19	57	149.15	14.7	1.18
20	60	157.00	14.8	1.18

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

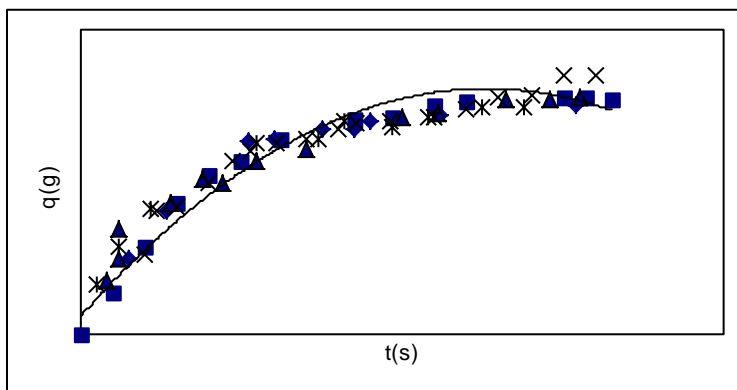


Fig. 3 General sedimentation curve

Measurements 1, 6, 7 and 10 are not reproducible due to a non-uniform distribution of the scattered particles obtained before the recordings (fig. 4a. and b.).

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.

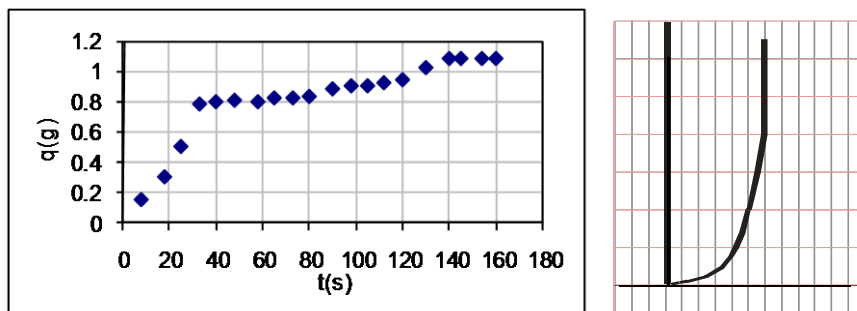


Fig. 4 Sedimentation curves using:
 a. $q(g) = f(t, s)$ dependence
 b. tensio-tixometer $q(mm) = f(t, min.)$ dependence

Table 3

Density of lignin measured by picnometer method

m_1 (g)	m_2 (g)	m_3 (g)	m_4 (g)
13.6512	14.6515	23.0630	22.4048
13.6512	14.6508	23.0622	22.4048
13.6512	14.6486	23.0642	22.4048

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.2), 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 (tab. 4).

Table 4

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

Time (s)	Sediment quantity (g)	Sedimentation rates (mm/s)
0	0	0.037
14	0.21	0.0165
30	0.63	0.0142
70	0.92	0.0034
80	0.97	0.0028
90	1.01	0.0018
100	1.02	0.0011
105	1.03	0.0140

Based on the resulted sedimentation rates, the particle radii of lignin were obtained (tab. 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	0.560	-
2	1.0 – 0.05	0.815	0.402	0.33 (0)
3	0.63 – 0.25	0.440	0.210	0.1572 (14)
4	0.25 – 0.20	0.225	0.1125	0.140 (30)
5	0.20 – 0.125	0.1625	0.0811	0.0741 (70)
6	0.16 – 0.10	0.130	0.0649	0.0628 (80)
7	0.10 – 0.09	0.095	0.0474	0.0523 (90)
8	0.09 – 0.08	0.085	0.0424	0.044 (100)
9	0.08 – 0.07	0.075	0.0369	0.038 (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.

REFERENCES

1. **Area M.C., Popa V.I., 2014** - *Wood Fibres for Papermaking*, Ed. Smithers Rapra, pp.. 106.
2. **Căprau Adina-Mirela, Ungureanu Elena, Popa V.I., 2009** - *Aspects concerning some biocides based on natural aromatic compounds and their copper complexes*, 15 th International Symposium on Wood, Fiber and Pulping Chemistry Oslo, pp. 197-201.
3. **Popa V.I., 2015** - *Wood bark as valuable raw material for compounds with biological activity*, *Celuloză și hârtie*, 64 (1), pp. 5-17
4. **Popa V.I., 2016** - *An example of biomass valorization as source of energy and chemical products*, *Celuloză și hârtie*, 65 (1), pp. 14-21.
5. **Ungureanu Elena, Dumitru Mariana, Tărnăuceanu Nicoleta, Nedelcu-Teodorescu Gabriela, 2006** - *Gravitational settling analyses applied to some natural zeolitic composite structures*, *Analele Științifice UIAC Iași, Seria Chimie*, XIV (2), pp. 129-134.
6. **Ungureanu Elena, 2011** - *Lignina, polimer natural aromatic cu ridicat potențial de valorificare*, Ed. PIM, Iași, pp. 112.