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Elaboration and Characterization of Self-Compacting Mortar Based Biopolymer

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Abstract**—**Lignin is a molecule derived from wood and also generated as waste from the paper industry. With a view to its valorization and protection of the environment, we are interested in its use as a superplasticizer-type adjuvant in mortars and concretes to improve their mechanical strengths. The additives of the concrete have a very strong influence on the properties of the fresh and / or hardened concrete. This study examines the development and use of industrial waste and lignin extracted from a renewable natural source (wood) in cementitious materials. The use of these resources is known at present as a definite resurgence of interest in the development of building materials. Physicomechanical characteristics of mortars are determined by optimization quantity of the natural superplasticizer. The results show that the mechanical strengths of mortars based on natural adjuvant have improved by 20% (64 MPa) for a W/C ratio = 0.4, and the amount of natural adjuvant of dry extract needed is 40 times smaller than commercial adjuvant. This study has a scientific impact (improving the performance of the mortar with an increase in compactness and reduction of the quantity of water), ecological use of the lignin waste generated by the paper industry) and economic reduction of the cost price necessary to elaboration of self-compacting mortars and concretes).

Keywords—Biopolymer, lignin, industrial waste, mechanical resistances, self-compacting mortars.

I. Introduction

MORTAR is a heterogeneous composite material. At the macroscopic scale, it has a cement paste structure composed of sand of various sizes, shape, pore, microcrack, and an interfacial transition zone localized around the sand grains. The resistance of mortars takes low values, which is reflected in cracks parallel to a horizontal plane different masonry units by accelerating the destruction of the latter [1]-[5].

Cement is regarded as the primordial element, which presents firstly a great potential link between the aggregates and masonry elements, and low flexural strength of the other part [2]. The search for solutions to improve microstructural properties is difficult in technological manufacturing processes, hence the need to explore other investigation means to overcome these difficulties.

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In the case of concrete, this problem is solved by the use of the armatures [6]. This allowed us to produce a possibility to apply a similar technique to reinforce mortars with a natural additive containing biopolymer.

In a context of increasing scarcity of fossil resources and preserving the environment, particular attention has been focused in recent years on the development of more respectful natural processes and the utilisation of renewable energy. Lignocellulosic polymers (cellulose, lignin, and hemicellulose) constitute an "inexhaustible" material source derived from the biomass [4].

Lignin is the second most abundant renewable polymer on earth after cellulose and they have over 70% of the total biomass. This work is focused on all of the structural features of lignin according to their nature and mode of extraction [3]. We will study thereafter lignin extracted from Aleppo pine by the process Klason, and spectroscopic technique (FTIR) will be used in order to establish a general structure of lignin of Aleppo pine and to show the various reactive functions that it possesses. This polymer has interesting physicochemical properties and presents an important development potential.

II. Experimental Study

A. Materials and Methods

The cement used in this study is a Portland Cement (CPJ CEM II / A 42.5). The sand used is a normal sand of the region Khemis Meliana (Algeria). Superplasticizers are tempo 12 (artificial (Polycarboxylate Type SIKA) and lignin (natural extract of Algeria Aleppo pine in the process Klason).

B. Extraction and Characterization of Lignin

The lignin used as a natural adjuvant in the mortar instead of commercial adjuvant is extracted from the wood of Aleppo pine which comes from the Algerian forest Bejaia, and after drying into the open air and at an ambient temperature, it is reduced to powder using a micro-mill and a sifting machine.

The powder obtained in the order of 0.8 mm (Fig. 1 (a)) is then treated in Soxhlet with toluene-ethanol (2-1, v/v) and then ethanol followed by water (Fig. 1 (b)). The parietal obtained residue is subsequently dried in the oven at a constant temperature of 28 °C [9].

The extraction of the lignin is made by the process without prehydrolysis Klason by similar protocol as that of Effland 1977 (Fig. 2). Test sample is 300 mg, weighed 0.1 mg, is carefully dispersed in sulfuric acid (H_2SO_4) 72%, where it is hydrolysed by sulfuric acid (H_2SO_4) at 72% for 2 h at 20±0.5 °C. After dilution with water to a content of 5% sulfuric acid

(H_2SO_4), the mixture obtained is posthydrolyzed by reflux under a gentle boil and periodic agitation for 3h.

(a) (b)

Fig. 1 Preparation of the material (a) Powder Aleppo pine, (b) Soxhlet extraction

The residue is collected by centrifugation or filtration (Fig. 3 (a)), then is washed to neutrality of the filtrate with deionized water, four times 50 ml, then dried in a dryingoven to constant weight and incinerated at 105 °C for 24h [8]-[10].

The yield of lignin obtained from the Aleppo pine wood is 29.5% of the initial mass.

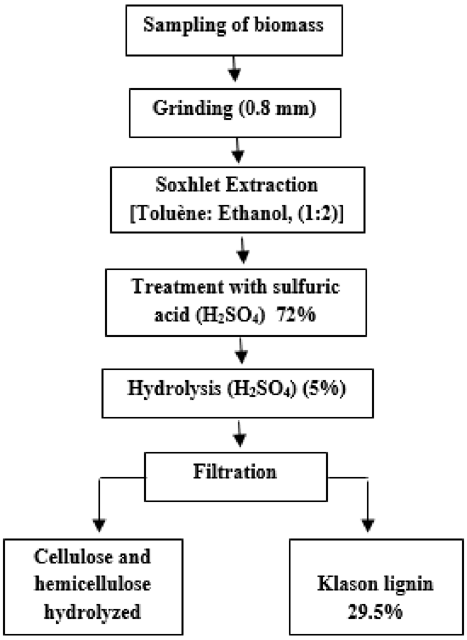

Fig. 2 Process for extracting the Klason lignin

After extraction of the Klason lignin, the residue obtained is in solid form (Fig. 3 (a)). For possible use as an adjuvant liquid, lignin must be dissolved in a suitable solvent. After many attempts to dissolve it in different solvents, we came to dissolve a certain quantity in NaOH solution (1.25%) with heating and stirring for a period of 4h (Fig. 3 (b)).

The FTIR analysis (Fig. 4) confirms the presence of the majority of the functional groups in the extracted lignin structure.

(a) (b)

Fig. 3 Lignin klason (a) solid lignin, (b) lignin solution

A broad band of 3400 cm^{-1} is mainly due to OH groups characteristics of polysaccharides. The band 1650 cm^{-1} corresponds to the absorbed water. We also note the presence of a band at 1450 cm^{-1}, which corresponds to CH, CH_2, and CH_3. The 1300 cm^{-1} band is a sign of cores G and S condensed [7].

With: G: leguaiacyl (2-méthoxy-phényl), S: lesyringyle (2,6-diméthoxyphényl), G, S are respective units of these aromatic alcohols (coniferylic and sinapyl) in the structure of lignin.

C. Elaboration of Different Formulations

The objective of this work is to study the effect of natural adjuvant of different percentages on the physicomechanical properties of mortars and to determine the best alternatives. These properties are compared with those of the mortar with the artificial superplasticizer (Tempo 12) [11]-[13].

TABLE I
DETAILS OF DIFFERENT FORMULATIONS

Constituent Mortar	Ciment (g)	Sand (g)	Water (ml)	Adj nat (ml)	Adjv TP_{12} (ml)
MT	450	1350	225	/	/
MT+TP_{12}	450	1350	180	/	8.1
MAN 5%	450	1350	171	9	/
MAN 10%	450	1350	162	18	/
MAN 20%	450	1350	144	36	/
MAN 30%	450	1350	126	54	/
MAN 40%	450	1350	108	72	/

with: MT: normal mortar, MT + TP12 (M12): mortar adjuvanted TEMPO12. MAN (5%, 10%, 20%, 30%, 40%): mortar with natural adjuvant.

III. Results and Discussions

In this part, we will present the tests carried out and the results obtained on the performance of different mortars based on natural (lignin) and artificial (Tempo12) adjuvants.

A. Physical Properties of the Studied Mortars

Spreading of Different Formulations: The workability in the fresh state is determined by the measurement of the spread of the mortar paste [11].

When increasing the percentage of natural adjuvant, spreading increases (Fig. 5), and the paste becomes much more fluid.

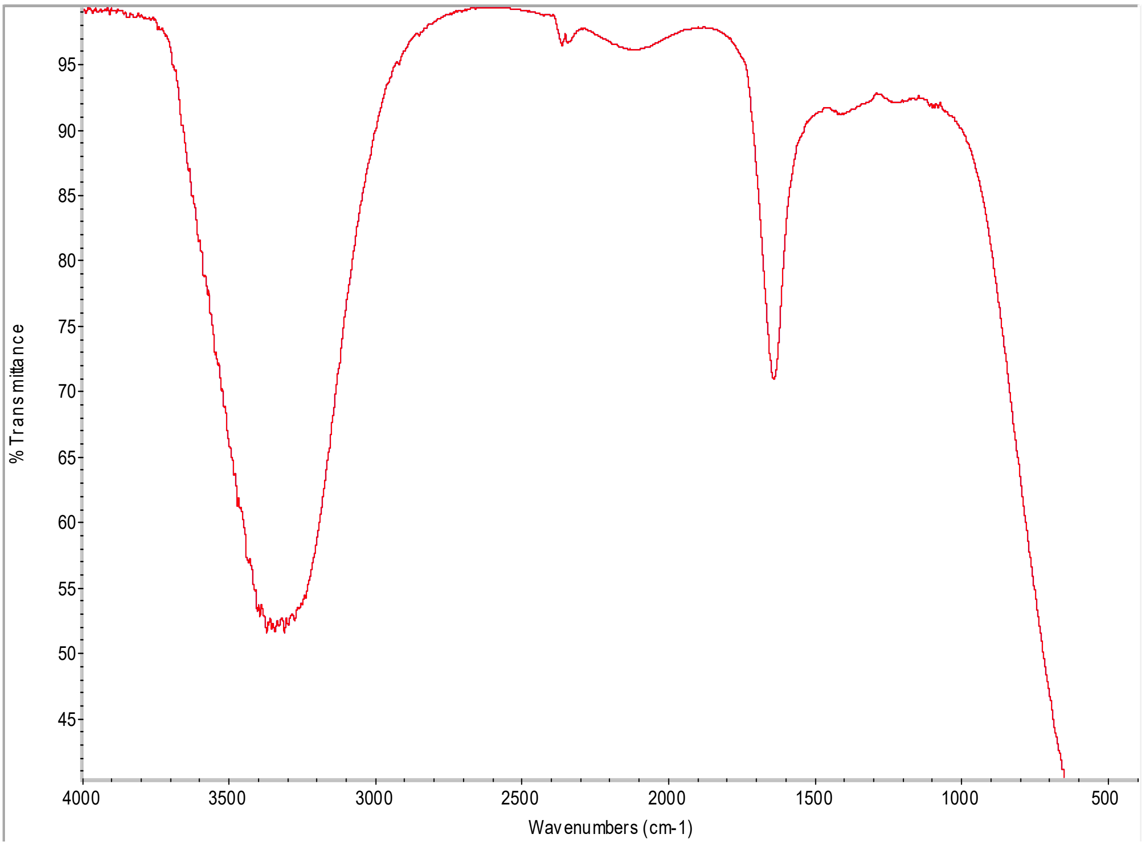

Fig. 4 FTIR analysis of lignin

Fig. 5 Spreading of different formulations

Density: This measure determines the volumetric efficiency (the mass ratio of the volume) of the composition of the fresh and hardened mortar, and checks the validity of the theoretical formulation.

In Fig. 6, we find that the density in the hardened state of the various mortars increases with the increase in the amount of natural adjuvant. For the same spread, we notice that the amount of commercial adjuvant used is 40 times greater than that of natural adjuvant. This is due to the effectiveness of natural adjuvant to be able to flocculate the cement particles to increase the fluidity and to facilitate hydration of the cement.

The densities of mortars made with natural adjuvant are larger than those tempo 12 mortars, especially for samples with a percentage of 30 and 40% of natural adjuvant. This is due to the decrease in porosity thus to increase the compactness of these mortars. The density is determined by:

$$\mathbf{Mv = M/V}$$

with: M = Mortar mass, V = Mold volume = 4x4x16 = 256 cm^3.

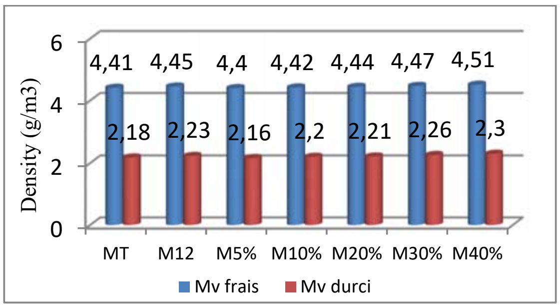

Fig. 6 Density in the fresh and cured state

B. Mechanical Properties of the Studied Mortars

Flexural and Compressive Strength: The bending tests are performed on the bending apparatus by placing the specimen symmetrical and centered on the plate of the hydraulic press provided with a three-point bending device, and a continuous load is applied to the specimen until the rupture. Fig. 7 shows the results for the flexural strength (after 28 days) of different mortars [12].

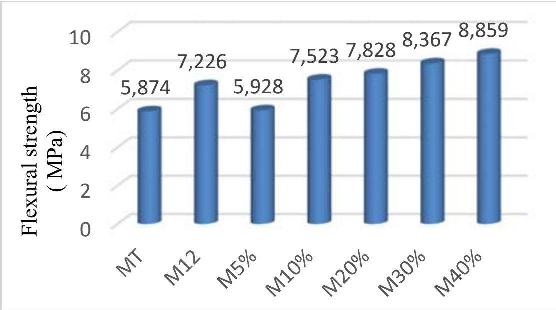

Fig. 7 Flexural strength

The compressive strength of the mortar was evaluated on the half-prisms from the three-point bending. The half prism is centered between the two plates of the apparatus, and a load is performed at a constant speed of 0.5 kN/s until failure. Fig. 8 shows the results for the compressive strength (after 28 days) of different mortars [12].

The main rule of the mechanical strength of mortar is directly related to the ratio W/C; that is to say, a low ratio leads to low porosity, therefore great compactness and consequently high mechanical strength.

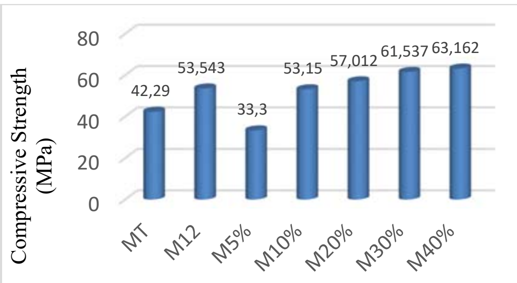

Fig. 8 Compressive strength

Figs. 7 and 8 represent the evolution of the mechanical resistance to compression and flexural of the various mortars. From these histograms, we note an increase in mechanical strength of mortars with the increased percentage of natural adjuvant, and this increase is due to the very effective action of this adjuvant that acts as a natural superplasticizier.

The presence of adjuvant in the cement matrix decreases the W/C ratio and increases the compactness and mechanical strengths of mortars.

IV. Conclusion

Lignin is a major constituent of lignocellulosic materials. The use of these resources is known at present as an undeniable resurgence of interest in the development of building materials.

This study focuses on the elaboration and characterization of self-compacting mortars based on a natural super plasticizer (lignin extracted from the Aleppo pine woods) and an artificial superplasticizer (Tempo 12 Polycarboxylates type SIKA) by decreasing the W/C ratio. This is to determine the quantity of optimal natural superplasticizer which will give the mortar the best physical and mechanical characteristics. Research undertaken in this study was satisfactory on the following points:

- The extraction of lignin by the Klason process is perfectly controllable.
- The analysis by FTIR confirms the presence of the majority of the functional groups in the structure of the extracted lignin.
- The use of the solubilized lignin extracted as an adjuvant to mortars is necessary and adequate for the manufacturing of self-compacting mortars.
- The performances of these mortars containing natural additive are improved by 20% compared to those of the mortars containing an artificial additive for an identical report W/C =0.4.
- The quantity of dry extract necessary in natural additive is 40 times smaller than that of the commercial additive for the elaboration of the mortars for a report W/C =0.4.

Thus, this study has impacts:

- *Scientific:* Improving physical and mechanical properties of mortars by using a biopolymer extracted from the Aleppo pine.
- *Economic:* Valorization of the Algerian wood and use of dry extract made of natural adjuvant with an amount of 40 times smaller than that of the commercial adjuvant.
- *Ecology*: Recovery of industrial waste for manufacturing paper.

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