The Canadian Society for Bioengineering
The Canadian society for engineering in agricultural, food, environmental, and biological systems.

La Société Canadienne de Génie Agroalimentaire et de Bioingénierie
La société canadienne de génie agroalimentaire, de la bioingénierie et de l'environnement

CSBE | SCGAB

Paper No. CSBE16-056

Potential use of Agricultural Biomass for Cement Composite Materials: Aptitude Index Development and Validation

M. Cabral[1], D. Zegan[2], J.H. Palacios[2], S. Godbout*[2], J. Fiorelli[1], H. Savastano[1], S. Gutierrez-Pacheco[2,3,4], R. Lagacé[4]

[1]Department of Biosystem Engineering, Sao Paulo University, Sao Paulo, Brazil
Avenida Duque de Caxias Norte, CEP 13635-900 Pirassununga, São Paulo, Brasil
Tel: +55-19-3565.4257, E-mail : matheusrc18@hotmail.com

[2]Research and Development Institute for the Agri-Environment (IRDA)
2700 Einstein Street, Quebec City, Quebec, Canada, G1P 3W8
Tel.:+1-418-643-2380, *E-mail: stephane.godbout@irda.qc.ca

[3]Soils and agri-food engineering department, Laval University
2425, rue de l'Agriculture, local 1122, Quebec City, Quebec, Canada, G1V 0A6
Tel.:+1-418 656-3145

[4]Civil and Agricultural Engineering department, Universidad Nacional de Colombia
Carrera 45 No 26-85, Edificio Uriel Gutiérrez, Bogota, Colombia
Tel.:+57-1-316-5000, E-mail: sgutierrezp@unal.edu.co

Written for presentation at the
CSBE/SCGAB 2016 Annual Conference
Halifax World Trade and Convention Centre
3-6 July 2016

ABSTRACT Several agricultural biomasses can be used as natural lignocellulosic particles for cement composites materials. However, not all of these biomasses have adequate aptitude for this application. To estimate the potential use of a particular biomass, an aptitude index (Ai) was developed and validated in this study. This study was conducted in three main steps: (a) review and selection of techniques to analyse agricultural biomass properties, focused on the potential use of lignocellulosic particles for use in cement-lignocellulosic particles composites, (b) aptitude index development, and (c) preliminary validation by analysing seven different biomasses. The results of this research can be exploited as a guideline for estimate the potential using of some agricultural biomass for cement-lignocellulosic particles composites.

Keywords: Composite, cement, lignocellulosic particles, biomass, residue, inhibition index, aptitude index.

INTRODUCTION To create value-added products in a sustainable way implies an efficient and responsible use of available resources. In the last decade, developments to get environmentally friendly and low-cost products for the building sector are becoming popular. Some studies have established the feasibility of using woody and other lignocellulosic components in building materials (e.g. cement-particleboards from lignocellulosic materials) (Bentur and Mindness, 2007; Brito et al., 2005; Matoski, 2005; Bilba et al., 2003; Giacomini, 2003; Iwakiri et al., 2000) replacing conventional fibers (Satyanarayana et al., 2007; Savastano et al., 2003; George et al., 2001).

The lignocellulosic particles-cement composites (LPCCs) are produced by mixing small pieces of lignocellulosic particles, cement, water, and in some cases, chemical additives have been applied to improve the setting time of cement. Cement-particle boards fabricated with lignocellulosic particles materials can be applied in a modular manner in walls, floors, roofs (Matoski, 2005), ceiling tiles, siding and roadside noise barriers. Structural uses include concrete-filled insulating forms.

Lignocellulosic particles-cement composites and concretes with lignocellulosic plants overcome the problems related to low tensile strength and strain capacities of plain concrete (Bentur and Mindness, 2007). In fact, fibers allow to increase into the composite material at least one of the following properties (Swift, 1981): (a) flexural strength, (b) post-crack load bearing capacity, (c) impact toughness, and (d) viscosity in the fresh state.

Cement-composites can be produced with inorganic reinforcement fibers (steel, glass, asbestos and synthetic) or natural reinforcement (organic particles). Composite materials with lignocellulosic materials are known to be biodegradable, low cost and to exhibit improved physical and mechanical properties in comparison to some other cement composites (Bentur and Mindness, 2007; Brito et al., 2005; Bilba et al., 2003; Giacomini, 2003; Iwakiri et al., 2000). The choice of natural particles has been motivated by the cost-benefit ratio and the ready availability of such materials.

Wood and timber industry residues have been the main sources of lignocellulosic materials in the industrial manufacturing of cement composite panels. However, some lignocellulosic residues studied as reinforcement in cement composites (include rice husk, coconut husk and sugarcane bagasse) have also adequate properties (Satyanarayana et al., 2007; George et al., 2001).

Some fiber species have adverse effects in LPCCs. In fact, the chemical composition of some lignocellulosic materials influences negatively the solidification of the cement retarding the hydration and setting time of the cementitious matrix. The biggest limitation to produce high quality LPCCs is the chemical incompatibility between the natural material and the binder as a result of the presence of some substances in the lignocellulosic materials which are extremely inhibitory for curing (Jefferson et al., 2004; Bilba et al., 2003. Such substances include sugars, extractives, hemicelluloses and lignin (Ferraz et al., 2012).

This work aimed to develop an approach for evaluating in a simple and a preliminary stage (before mechanical behavior tests) the potential of an agricultural lignocellulosic material as constituent in lignocellulosic particles-cement composites. The developed approach should avoid costs, material and time required by mechanical behavior tests.

MATERIALS AND METHODS

Step 1: Review and techniques selection An exhaustive review of techniques for evaluating lignocellulosic material for cement composites materials was carried out (Cabral et al., 2015). From this review, two techniques were chosen: chemical characterization (for cellulose, hemicellulose, lignin and extractives content) and thermometry analysis. These techniques were chosen because of their relatively low complexity, quick and low cost analysis. Efforts of this research work were then focused the development of a unique aptitude index as an easy-get parameter for evaluating the behavior of a lignocellulosic material in LPCCs.

Thermometry analysis, as proposed by Hofstrand et al. (1984), assesses compatibility of lignocellulosic particles and cement by the hydration temperature method coupled to calculation of an inhibition index (I). In this analysis, the inhibition index can be computed from the maximum temperature of hydration, the time required to reach the temperature and the maximum slope of the exothermic curve of the lignocellulosic particles–cement-water mixture when compared respectively with the values of the uninhibited cement (Eq. 1).

$$\mathrm{I} = 100 * \left(\frac{\mathrm{T}-\mathrm{T}`}{\mathrm{T}}\right)\left(\frac{\mathrm{H}`-\mathrm{H}}{\mathrm{H}}\right)\left(\frac{\mathrm{S}-\mathrm{S}`}{\mathrm{S}}\right) \quad (1)$$

where I is the inhibition index (%);T is the maximum temperature of cement/water mixture (◦C), T′ is the maximum temperature of fiber/cement/water mixture (◦C); H is the time to reach maximum hydration temperature of cement in cement/water mixture (h); H′ is the time to reach maximum hydration temperature of cement mixture in the fiber/cement/water mixture (h); S is the maximum temperature increment of the curve in the cement/water mixture (◦C/h); S′ is the maximum temperature increment of the curve in the fiber/cement/water mixture (°C/h). A low I-value indicate a high compatibility (Table 1) (Okino et al., 2004; Hachmi et al., 1990).

Table 1 Inhibition index used to classify fiber compatibility on LPCCs

Inhibition index I (%)	Grading
0-10	Low inhibition
10-50	Medium inhibition
50-100	High inhibition
> 100	Extreme inhibition

Step 2: Aptitude index development and utilization The aptitude index must preliminary indicate if the high amount of dissolved material extracts (e.g. hemicellulose) denotes interference in cement curing. In this study, it was proposed to develop an aptitude index (*Ai*) based on the relationship between inhibition index (*I*, %) and the chemical characteristics (cellulose,

hemicellulose, lignin or extractive) of a biomass (*m*, %) (Eq. 2 and 3). If this correlation is established, it is possible to determine the *Ai* or *I* in a preliminary manner to confirm the compatibility into the cement, without a thermometry analysis.

$$I = f[m] \qquad (2)$$

$$Ai = \frac{1}{I} = f[m] \qquad (3)$$

Step 3 : Validation of the aptitude index

Lignocellulosic materials

The aptitude index value was determined for seven agricultural lignocellulosic materials. Chemical characterization and thermometry analysis from each material were combined to develop the proposed parameter. Lignocellulosic materials selected from Canadian agriculture included: (a) agricultural residues: (1) oat straw, (2) Jerusalem artichoke stalk, and (3) corn stalk; and (b) agricultural biomass: (4) willow, (5) switchgrass, (6) miscanthus, and (7) hemp straw. All lignocellulosic material was dried and milled (Wiley Mill Model 3, Thomas Scientific, Swedesboro, NJ, USA) until particles passed through the miller plate of 8 mm openings. To refine the operation, the material was sieved. Particles obtained from 8 and 10 mm mesh were collected for this study.

Chemical Characterization

The methodology proposed by the French Association of Normalization (Afnor, 1993) was followed to determine the cellulose, hemicellulose and lignin content from lignocellulosic materials. The method described by ASTM D – 1110 (1994) was followed to determine the soluble extractives content.

Thermometry analysis The method described by Hofstrand et al. (1984) and Okino et al. (2004) was followed. For each lignocellulosic material, 15 g were mixed with 200 g of High-Early Portland cement (Type III and type HE acording to CSA and ASTM C150, respectively), and a specific quantity of water for each sample calculated by equation 4.

$$W = C\left[LM\left(0.3 - \frac{HD}{100}\right)\right] \qquad (4)$$

Where *W* is the water volume (ml), *C* is the amount of cement (g), *LM* is the amount of lignocellulosic material (g) and *HD* is the water content in each lignocellulosic material (%, d.b). The mixing time did not exceed five minutes. Immediately after, the mixture was transferred to plastic bags (19.7 x 28.6 cm). Additionally, a cement-water control sample was prepared without lignocellulosic material. Each sample was put into a 500 ml stainless steel thermos isolated by a vacuum double wall. All thermos were put into a 45.4 L cooler (Coleman, Wichita, Kansas, USA) filled with fiber glass insulation (R-40 EcoTouch, Toledo, Ohio, USA). Temperature in each sample was followed by a type “J” thermocouple (Omega, Stamford, Connecticut, USA) inserted through the top of the thermos. Thermocouples were connected to a data acquisition system (Brand Campbell Scientific Data 21X) which ensured temperature data recording every minute during 24 hours. This test was repeated three times.

RESULTS AND DISCUSSION Thermometry analysis results (Table 2) showed that evaluated lignocellulosic materials have a low or a medium inhibition according to Table 1. Similar I-values were found by Aamr et al. (2008) to cement-flax waste particles mixture (flax particles: 5 % of weight content). The particles-cement mixtures that showed lowest inhibition rates (< 5 %) were willow, hemp straw, Jerusalem artichoke stalk and corn stalk. The highest maximum temperature values (63.3-70.2 °C) and the lowest times to reach maximum temperature (9.4-9.9 h) were obtained from these four materials. Linear proportionality between compressive strength and the maximum hydration temperature was observed by Lee and Hong (1986). However, Blankenhorn et al. (1994) research indicated that as hydration time decreases, compressive strength decrease.

Table 2. Thermometry analysis results of evaluated lignocellulosic materials

Lignocellulosic material	**T' (°C)**	**H' (h)**	**S' (°C/h)**	**I (%)**
Switchgrass	49.2	10.6	4.7	12
Miscanthus	46.1	13.0	3.5	27
Oat straw	46.1	13.6	3.4	30
Jerusalem artichoke stalk	66.6	9.9	6.8	4
Hemp straw	64.8	9.4	6.9	3
Willow	70.2	9.4	7.5	2
Corn stalk	63.3	9.8	6.5	5
Cement	86.5	7.2	12.1	-

These four materials having the lowest *I*, have also in common the lowest hemicellulose contents (< 20 %) according to Table 3. Hemicellulose may be degrading into simple sugars such as glucose, mannose and xylose. These simple sugars are responsible of cement inhibitory effect in setting process. Carbohydrates react with calcium, aluminum and iron cations (contained in cement) thus reducing the crystalinity, the mixture resistance and the cement hydration rate (Souza, 1994). It can be observed that the higher the hemicellulose content, the greater the inhibitory effect on cement setting. Any other chemical characteristic seems to have a correlation with the inhibition index.

Table 3. Chemical characterisation of the seven lignocellulosic materials studied

Lignocellulosic material	**Lignin (%)**	**Cellulose (raw) (%)**	**Hemicellulose (%)**	**Extractives (%)**
Switchgrass	24.0	40.9	26.1	16.5
Miscanthus	24.3	41.8	23.8	25.3
Oat straw	24.0	38.5	20.3	18.4
Jerusalem artichoke stalk	21.8	49.4	13.3	31.0
Hemp straw	26.7	54.5	10.8	8.4
Willow	58.0	53.5	7.1	11.2
Corn stalk	26.8	44.2	18.9	13.2

The correlation between Inhibition index I and the hemicellulose content for the seven evaluated lignocellulosic materials is showed in Figure 1. This figure shows an apparent correlation between hemicellulose content and inhibition index, but only for lignocellulosic materials having low inhibition index (Fig. 2) (R^2=0.99). The correlation is lost when medium inhibition materials are included. The aptitude index could be then obtained from hemicellulose content.

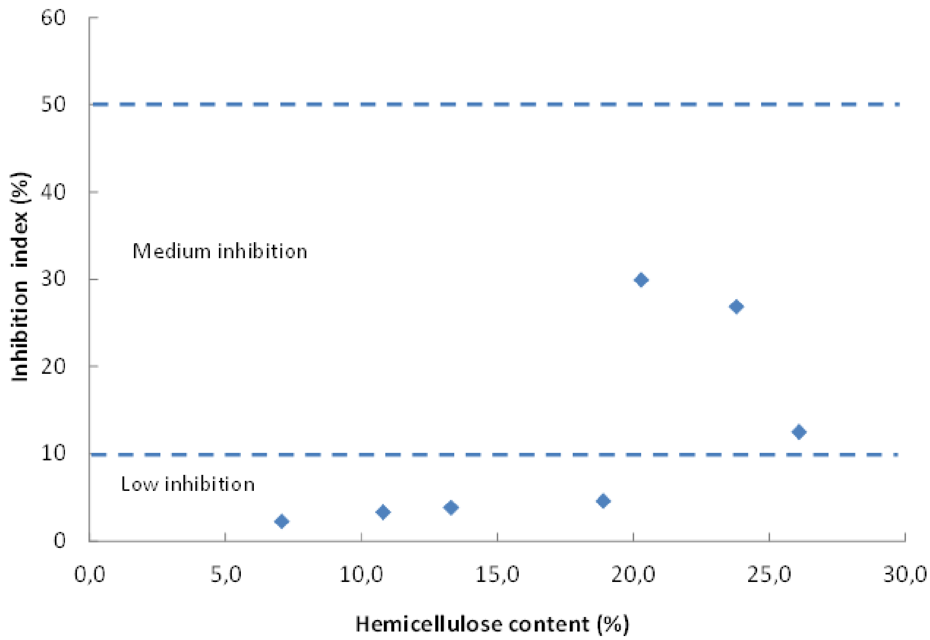

Figure 1: Inhibition index I and hemicellulose content relation for the evaluated lignocellulosic materials

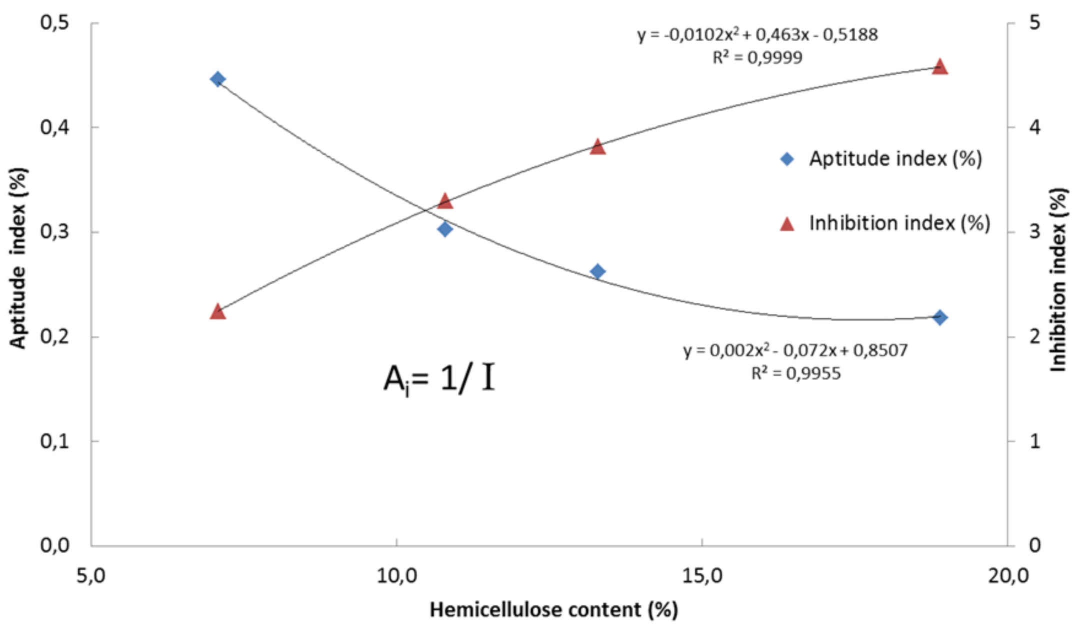

Figure 2: Relationship between hemicellulose content with Aptitude Index (Ai) and Inhibition index (I) for the evaluated lignocellulosic materials

Before carrying out a thermometry analysis, the aptitude index (Ai), and hence the potential of a lignocellulosic material for use in LPCCs, can be estimated by the hemicellulose content (Hc) from the chemical characterization by the following equation:

$$Ai = 0.002 \cdot Hc^2 - 0.072 \cdot Hc + 0.8507 \qquad (5)$$

This approach can be applied to lignocellulosic materials having low hemicellulose contents (< 20%). In the case of high cellulose content, the possibilities of using the material as it in FCCs are low. It should be recommended to treat it.

CONCLUSIONS AND RECOMMENDATIONS An index to evaluate the potential of an agricultural biomass or residue in cement composite materials (LPCCs) was developed using only two simple methods: chemical characterization and thermometry analysis. Both tests have a relatively easy execution and results allow knowing the performance in LPCCs. The aptitude index developed, which defines the compatibility lignocellulisic material-cement, is based on the relationship between the hemicellulose content of the particle and the inhibition index. This technique allows, to estimate in a preliminary manner, the potential use of a particular biomass for the cement composites materials, only with chemical characterization. If hemicellulose content < 20 %, aptitude index can be applied. Otherwise, pretreatment of material is recommended before use in LPCCs, or rejecting it. The results of this research could be used as a guideline for using agricultural residues as LPCCs. However, further tests should be carried out in order to validate and refine the approach.

Among the seven biomass evaluated, willow, Jerusalem artichoke, hemp stalk and corn stalk showed an important compatibility with the Portland cement: low inhibition rates, I< 5 % and low amount of hemicellulose. Further should study the relation of the aptitude index with the mechanical resistance of the composite materials.

REFERENCES

Aamr-Daya, E., Langlet, T., Benazzouk, A., Quéneudec, M. 2008.Feasibility study of lightweight cement composite containing flax by-product particles: Physico-mechanical properties, Cem. Concr. Compos., 30(10), 957–963.

Afnor- NF V03-040. 1993. Produits agricoles et alimentaires - détermination de la cellulose brute - méthode générale. Association Française de Normalization, Saint Denis la Plaine, France (www.afnor.fr).

Bentur, A., Mindness, S. 2007.Fiber reinforced cementations composite, second ed., Taylor & Francis, Abingdon.

Bilba, K., Arsene, M.-A., Ouensanga, A. 2003. Sugar cane bagasse fiber reinforced cement composites. Part I. Influence of the botanical components of bagasse on the setting of bagasse/cement composite, Cem. Concr. Compos. 25, 91–96.

Brito, et al.2005. Chapas de madeira aglomerada de uma camada de Pinus elliottii Engelm com a adição das cascas de Eucalyptus pellita F. Muell. Cerne. 11, 369-375.

Cabral, M.R., Palacios, J.H., Godbout, S., Lagacé, R., Fiorelli, J., Savastano, H., Zegan, D. Development of an indicator to evaluate the potential use of agricultural wastes and biomasses for the production of cement composite materials. Part 1: Literature review and set-up of the methodology. In Proceedings of CSA Joint International Conference, 8th International

Symposium on Cement Based Materials for a Sustainable Agriculture. Iasi, Romania, October 22-25th, 2015.
Ferraz, J.M., Del Menezzi, C.H.S., Souza, M.R., Okino, E.Y.A., Martins, S.A. 2012. Compatibility of Pretreated Coir Fibers (Cocos nucifera L.) with Portland Cement to Produce Mineral Composites, International Journal of Polymer Science 2012, 1 – 7.
George, J., Sreekala, M. S., Thomas, S. 2001. A review on interface modification and characterization of natural fiber reinforced plastic composites, Polym. Eng. Sci. 41, 1471–1485.
Giacomini, N.P. 2003. Compósitos reforçados com fibers naturais para a indústria automobilística. Thesis (Masters degree) – Escola de Engenharia de São Carlos/Instituto de Física de São Carlos/Instituto de Química de São Carlos, Universidade de São Paulo, São Carlos, 168 p.
Hachmi, M., Moslemi, A.A.,Campbell, A.G. 1990. A new technique to classify the compatibility of wood with cement, Wood Sci Technol. 24, 345–354.
Hofstrand, A.D., Moslemi, A.A., Garcia, J.F. 1984. Curing characteristics of wood particles from nine northern Rocky Mountain species mixed with Portland cement, Forest Prod. J. 2, 57–61.
Iwakiri, S., Cunha, A.B., Alburquerque,C.E.C., Gorniak, E., Mendes, L.M. 2000. Resíduos de serrarias na produção de painéis de madeira aglomerada de eucalipto. Revista Scientia Agrária. 1, 23-28.
Jefferson, P.G., Mccaughey, W.P., May, K., Woosaree, J., Mcfarlane, L. 2004. Potential utilization of native prairie grasses from western Canada as ethanol feedstock, Canadian J Plant Science 84, 1067–1075.
Matoski, A. 2005. Utilização de pó de madeira com granulometria controlada na produção de painéis de cimento-madeira. Thesis (Doctoral), Universidade Federal do Paraná, Curitiba, 202 p.
Okino, E.Y., Souza, M. R. d., Santana, M. A., Alves, M. V. d. S., De Sousa, M. E., Teixeira, D. E. 2004. Cement-bonded wood particleboard with a mixture of eucalypt and rubberwood, Cem. Concr. Compos. 26, 729–734.
Satyanarayana, K. G., Guimarães, J. L., Wypych, F. 2007. Studies on lignocellulosic fibers of Brazil. Part I: Source, production, morphology, properties and applications, Compos. Part A Appl. Sci. Manuf. 38, 1694–1709.
Savastano, H., Warden, P., Coutts, R.S. 2003. Potential of alternative fiber cements as building materials for developing areas, Cem. Concr. Compos. 25, 585–592.
Souza, M.R. 1994. Durability of cement-bonded particleboard made conventionally and carbon dioxide injection, Thesis (Doctoral), University of Idaho, Idaho, 123 p.
Swift, D.G. 1981. The use of Natural organic fibers in cement: some structural considerations in: I.H. Marshall (Eds.), Composite structures, Applied science publisher, London, 1981, pp. 602-617.