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An Alternative Modeling Equation to Estimate Wood-Cement Inhibition as Function of Wood-to-Cement Ratio

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Abstract

An alternative formula to estimate wood-cement inhibitory factor (I_f) is presented, integrating the total released heat and the residual cooling heat recorded at 24h hydration limit, and weighted to the wood-to-cement ratio (WCR) and to the time to reach maximum heat released by the second cement reaction. Five different WCRs were tested based on 3, 7, 11, 15 and 20g of wood provided from 12 different Moroccan biomass species. According to the ANOVA results, there is no significant difference between the newly obtained I_f-value and the I_d-value deduced from the previously developed modeling C_s-index equation. The two extreme inhibition classification classes were defined: upper class (I_f > 95%), and lower class (I_f < 5%). The analysis of the WCR parameter effect on I_f-value showed the existence of different evolution trends: linear, logarithmic, polynomial and puissance curve shape, depending on wood species. This I_f-approach is compared with other inhibitory indices cited in the literature.

Keywords: Hydration inhibition factor, hydration heat profile, wood-cement ratio, inhibition classes.

INTRODUCTION

In the context of the strategies undertaken globally to stimulate the green economy and to protect our environment, many investigations were undertaken to upgrade any lignocellulosic waste that contributes to the environment pollution. Invaluable small woods as well as industrial wood residues could be suitable for manufacturing wood-cement (WC) composites as building materials [1-3], cement particleboard [4,5], or as wood wool cement board [6]. However, the natural incompatibility observed between natural lignocellulosic materials and cement must be overcome in WC composites.

The cement hydration process undergoes two exothermic reactions in aqueous mixtures. Calorimetric measurements show that the plots 'released heat vs. hydration time' are not uniform and that wood particles reduce usually the heat released; this property is wood-species dependent. Various methods were developed to estimate WC inhibition. The first inhibitory index was developed by Weatherwax and Tarkow [7] based only on the maximal time (t_{max}) required to reach the maximum temperature (T_{max}). Since this approach is lacking of consistency, Hofstrand et al. [8] attempted to find a global inhibitory index by combining T_{max} and the maximum slope (S_{max}) of plots 'T *vs.* t'. Despite their simplicity, these two both approaches are still not convincing, especially in cases of incompatible wood species, which result in index-values > 100%, or in case of accelerator woods that would give index-values < 0. Pereira et al. [9] developed another inhibitory index, $I^{+}_{(T,t,S)}$, based on an average of the three parameters indicated in the lower case letters. In the same way, Olorunnisola [10] suggested also the time ratio as a parameter to estimate WC inhibitory index-value. However, these two last inhibitory indices were found to be significantly different from the Id-value deduced from the previously developed modeling C_s-index equation [11].

As indicated above, more research is needed to understand better the wood species inhibition as function of the WCR variation and also to confirm or reject the Id-value deduced from the absolute C_s-value based only on the hypothesis principle of $I_d + C_s = 1$ [11].

MATERIALS AND METHODS

In this study, 12 biomass species were collected from different forest areas of Morocco (Table 1). The samples were fragmented, crushed, sieved, and naturally dried in the open air. Only a fraction of the particles passing through a 20 mesh screen and retained by the 40 mesh screen was tested. The moisture content was determined to calculate the amount of distilled water needed to add to the mixture with CPJ45-Portland cement (Holcim Factory, Morocco). This mixture consisted of 15 g of dry wood, 200 g of cement and 90.5 g of distilled water [7]. For the neat cement (NC), the proportion was 80 g water and 200 g cement. The water/wood ratio was

0.7; thus, 10.5 g of water was mixed with 15.0 g dry wood [12]. Four other wood masses were tested: 3, 7, 11, and 20 g of any studied wood species. All these hydration tests were performed in three replicates in Dewar flasks [13] equipped with K-type thermocouples connected to a programmable temperature recorder. The measured temperatures (T) were corrected by the room temperature (rT), i.e. as T minus rT, and then converted into heat per gram (J/g) based on the following thermal capacity constants of the mixture ingredients: cement (0,778 J/°C.g), water (4,184 J/°C.g), and wood (1,356 J/°C.g). From statistical point of view, variance analyses (ANOVA I) were applied based on one factor (inhibition estimation methods).

Table 1: Different Moroccan heartwood species and the grass *Stipa tenacessima* L. submitted to hydration testing with cement.

Scientific name	Morocan forest area
Acacia mearnsii De Wild.	Maâmora
Argania spinosa L.	Admine
Eucalyptus gomphocephala DC.	Maâmora
Quercus ilex L.	Oulmès
Quercus suber L.	Chefchaouen
Quercus suber L.	Maâmora
Pinus brutia Ten.	Tetouan
Pinus halepensis Mill.	Chefchaouen
Pinus halepensis Mill.	Oulmès
Pinus pinaster Lam.	Kénitra
Pinus pinea L.	Tetouan
Stipa tenacessima L.	Bouarfa

RESULTS AND DISCUSSION

The new inhibitory equation formula

The aim of this study is to propose an alternative inhibitory factor (I_f) that would estimate WC inhibition basically using the concept how much a WC heat curve deviates from that of the NC control. This investigation was undertaken in order to treat any heat curve profile with two maxima (2 Q_{max}) (Figure 1) or with a single Q_{max} (1 Q_{max}). This approach would let a hydration period of 24 h to categorize any WC-hydration curve profile without exception. This Figure 1 illustrates the cement hydration process that goes through two exothermic reactions. The first reaction (phase 1) is characterized by its maximum heat: Q_1' for NC and Q_1 for WC, reached at their equivalent time t_1' and t_1, respectively. This first maximum heat is followed by a cooling period characterized by its decrease to reach a minimum cooling heat (Q_{c1}) reached at a dormant time (t_d). The magnitude of this dormant period is directly wood species dependent. The second exothermic reaction (phase 2) is characterized by its maximum heat: Q_2' for NC and and Q_2 for WC, and by their equivalent time t_2' and t_2, respectively. This second maximum heat is followed by a heat cooling phase to reach finally a residual cooling heat level (Q_{cr}) recorded at 24 h. The Q_{cr}-value would somehow characterize the final heat evolution state depending on type of wood species particles incorporated into mixture. It appears from this figure that the WC hydration heat curve is clearly delimited by the previously defined heat values levels: Q_1, Q_{c1}, Q_2 and Q_{cr}.

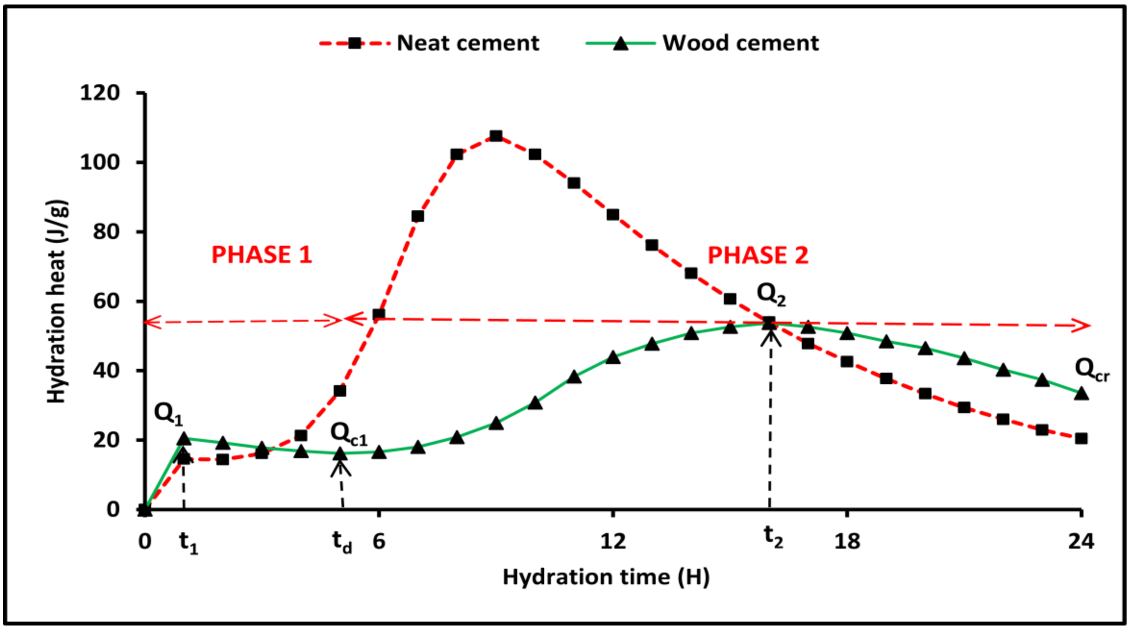

Figure 1. The important points characterizing the wood-cement heat evolution observed during the 24 h hydration period.

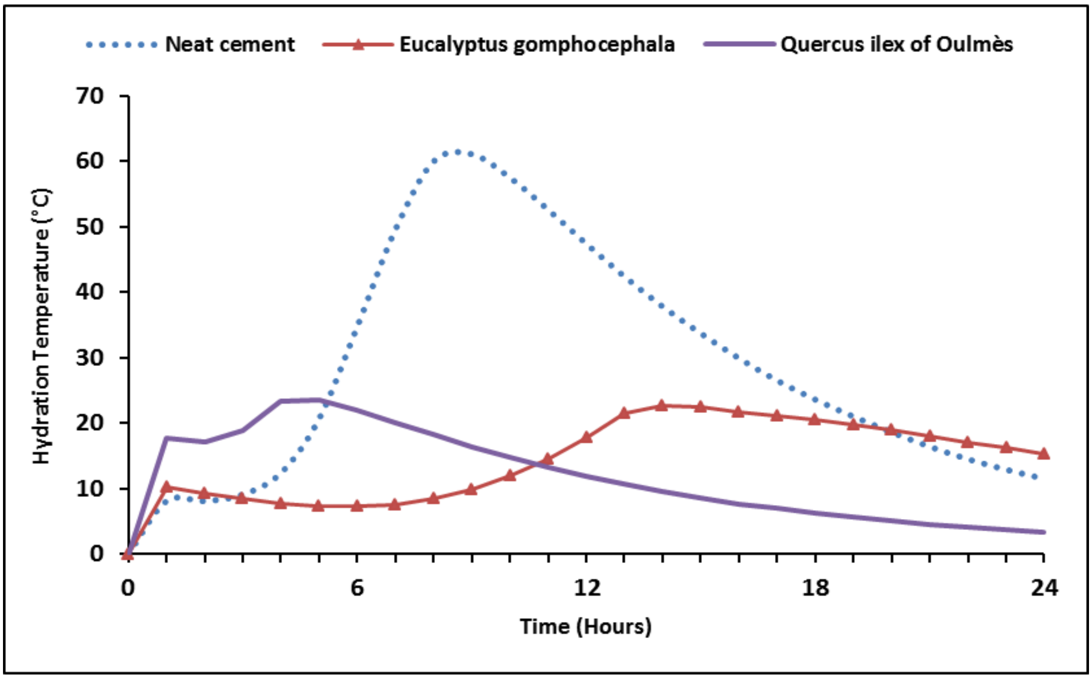

Figure 2. Examples of wood that accelerates (*Quercus ilex* L. of Oulmès) and wood that delays (*Eucalyptus gomphocephala* DC.) the 2nd cement exothermic reaction.

Therefore, the positioning level of the curve with respect to the Y-axis (J/g) is automatically determined by the values that will be recorded for these four heat levels. The incorporation of wood particles into mixture would disrupt the cement exothermic reactions, decreasing then heat at lower levels depending on wood species type. Given this observed effect, it appears necessary to estimate WC-heat curve deviation from that of the NC control by considering the following I_f-factor that integrates in a first step all the four equivalent heat levels as follows:

$$If = \frac{[Q'_1+(Q'_2-Q'_{c1})+Q'_{cr}]-[Q_1+(Q_2-Q_{c1})+Q_{cr}]}{[Q'_1+(Q'_2-Q'_{c1})+Q'_{cr}]} \quad (1)$$

With Q_1 : Maximum heat (J/g) of the first reaction; Q_2: Maximum heat (J/g) of the second reaction; Q_{c1}: Minimum cooling heat reached after the first reaction; Q_{cr}: Residual cooling heat (J/g) recorded at 24 h. Variables with apostrophe (') represent NC control

However, it is possible that two wood species may have the same heat levels with respect to the Y-axis but at different positions with respect to the hydration time reported on the X-axis. Figure 2 illustrates an example of a real case where the *Quercus ilex* wood of Oulmès is considered as wood that accelerate the second reaction while the *Eucalyptus gomphocephala* wood tends to delay it [14].

Therefore, the positioning with respect to the X-axis (Hydration time) as well as the WCR variable appear to be important parameters to be considered as elements that would help to better discriminate between wood species. And the best way to deal with is to integrate into the equation (1) an adjustment coefficient that would characterize more the deviation effect observed between the WC heat curve profile and that of the NC control as follows:

$$If = \frac{[Q'_1+(Q'_2-Q'_{c1})+Q'_{cr}]-[Q_1+(Q_2-Q_{c1})+Q_{cr}]}{[Q'_1+(Q'_2-Q'_{c1})+Q'_{cr}]} \times \left[1+\left(\frac{W}{C}\right)\times Exp\left(\frac{t_2-t'_2}{t_2+t'_2}\right)^2\right] \quad (2)$$

With W : Wood mass (g) incorporated into cement mixture; C: Cement mass (g) ; t_2: Time (hour) to reach the maximum heat (J/g) of the second reaction. Variables with apostrophe (') represent NC control.

For a first practical validation, this I_f-formula was applied on four hydration replicates done on Moroccan Portland cement. The replicate whose maximum heat is the highest one was selected as the replicate control (RC). The curves of the other replicates were then compared respectively to that of RC. Data for this comparison as well as their I_f -values are shown in Table 2. The application of this formula on the RC itself confirmed that its hydration heat curve is 100% similar to itself with an I_f-value of 0% inhibition. It appears that the hydration heat curves of the cement replicates are similar to each other; since their I_f-value varies from a minimum of 0% recorded for the RC control to a maximum of 4% for the second replicate (Table 2).

Table 2: Characteristics of the heat curves obtained from several hydration replicates applied on Moroccan CPJ45-cement type. The replicate whose maximum heat is the highest one was selected as the replicate control for the remaining three replicates.

Moroccan cement	Q_1' (J/g)	Q_{c1}' (J/g)	Q_2' (J/g)	Q_{cr}' (J/g)	t_1' (h)	t_2' (h)	I_f-value (%)
Replicate control	12,78	12,08	82,66	39,23	1	14	0,00
Replicate1	10,51	9,98	77,58	40,63	1	15,5	3,14
Replicate2	13,31	10,33	75,66	39,05	1	15	4,00
Replicate3	12,26	11,03	80,56	37,83	1	14	2,43

If all the hydration testing replicates are carefully performed, also their I_f-values will be all less than 5%. This 5% limit could be considered as the high borderline of the lowest inhibition class (I_f < 5%) reserved for hydration heat curves that more or less perfectly fit in shape one of the heat curves of the NC replicates. As result, this lower inhibition class would constitute the threshold class that is impossible for any wood-cement I_f-value, obtained on 15g wood basis, to belong to it.

Assessment of wood species inhibitory factors

The new I_f-values obtained for the 12 studied wood species at different WCRs as well as those of their deviation index (deduced I_d-values) are reported in table 3 for comparison purposes. Note that the least inhibitory wood species is *Pinus pinaster* Lam. with minimum I_f-values (4,2% for WCR=3/200 – 50,9% for WCR=20/200) and the most inhibitory one is *Argania spinosa* L. with maximum I_f-values (60,4% for WCR=3/200 – 100,6% for WCR=20/200). At first glance, the values obtained for the I_f-factor appear slightly different from those of the I_d-index (Table 3).

Table 3: Average data characterizing WC inhibition of different types of biomass, as estimated at different wood-to-cement ratios by the I_f-factor and I_d-index.

Scientific name	Moroccan forest area	3g		7g		11g		15g		20g	
		I_d	I_f	I_d	I_f	I_d	I_f	I_d	I_f	I_d	I_f
Pinus pinaster Lam	*Kénitra*	4,4	4,2	10,4	7,1	12,8	9,8	18,3	17,2	42,6	50,9
Pinus pinea L.	*Tetouan*	7,3	1,4	11,6	12,5	21,9	16,1	28,1	31,4	61,0	74,2
Pinus halepensis Mill.	*Chefchaouen*	5,0	7,5	12,8	12,2	21,5	18,1	29,7	31,6	68,1	83,4
Pinus brutia Ten.	*Tetouan*	7,0	10,2	18,4	16,6	27,4	29,9	37,3	53,2	59,2	84,1
Eucalyptus gomphocephala DC.	*Maâmora*	5,8	5,2	18,3	8,8	26,7	12,7	43,4	36,9	58,7	83,6
P. halepensis Mill.	*Oulmès*	5,8	6,2	12,3	17,9	28,8	23,7	45,4	52,4	69,7	86,9
Quercus suber L.	*Chefchaouen*	8,1	8,7	17,3	19,9	33,2	40,9	55,2	66,2	71,0	79,7
Quercus ilex L.	*Oulmès*	16,4	11,4	32,6	29,4	47,2	59,8	70,2	71,2	87,8	86,0
Q. suber L.	*Maâmora*	36,8	50,8	69,6	76,5	85,2	84,3	90,5	89,7	94,0	94,7
Acacia mearnsii De Wild.	*Maâmora*	27,6	18,0	52,8	74,5	66,5	85,6	89,6	88,1	93,6	94,2
Stipa tenacissima L.	*Bouarfa*	37,7	34,7	60,9	83,0	83,7	88,6	91,6	91,8	92,6	95,4
Argania spinosa L.	*Admine*	51,1	60,4	75,5	81,7	90,1	90,4	95,7	99,5	96,4	100,6

However, the results of ANOVA (Table 4) show that there is no significant difference between this new I_f-factor method and that of the I_d–value deduced from the modeling C_s-formula developed by Hachmi et al. [11]. This result confirms the hypothesis assumption proposed by these authors ($I_d + C_s = 1$).

The variation of the WCR significantly inhibits the cement exothermic reactions. In fact, the I_f-values increase with this WCR parameter as show their curves obtained for the 12 studied wood species in Figure 3.

Table 4: Analysis of variance between the newly proposed I_f-factor method and that of the I_d –value deduced from the Cs-modeling equation.

Source of variation	Df	Squares Sum	Mean	F	Pr > F
Model	1	1228,403	1228,403	1,167	**0,281**
Residual	358	376928,633	1052,873		
Total	359	378157,036			

Df: Degree of freedom; F: calculated Fischer-test; Pr>F: Probability that the F-Table is greater than the calculated F.

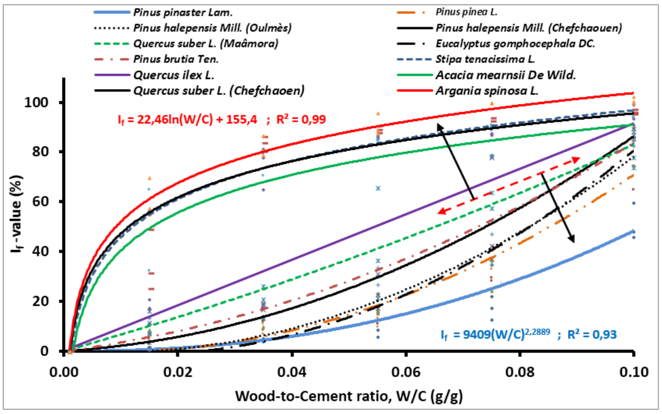

Figure 3. The evolution of the I_f-value observed for 12 Moroccan wood species as function of the wood-to-cement ratio.

Note that these curves present different tendencies: logarithmic curve shape for the inhibitory wood species characterized by 1 Q_{max} heat curve profile such as *Argania spinosa*; linear or polynomial equation curves for certain number of other wood species with 2 Q_{max}, and power tendency for the least inhibitory ones such as *Pinus pinaster*. The 12 curves shown in this figure 3 appear to be symmetrically distributed on both sides of the diagonal line, from which the trend of the curves changes: upward, from the linear to the logarithmic profile of *Argania spinosa*, and downward, from the linear to polynomial and power profiles. These different inhibition behaviours observed for these wood species as function of WCR may later constitute an interesting basis for determining the classification of the WC inhibition into different classes. In fact, these different I_f-value evolution trends appear somewhat to confirm the three WC compatibility classes proposed by Hachmi and Moslemi (1989) [15]: class of incompatible wood species (Ca ≤ 28%) could correspond to heat curves of logarithmic tendency, class of moderately compatible species (28% < Ca ≤ 68%) to the linear trend curves, and compatible species class (Ca > 68%) to the polynomial and power trend ones. But for better clarification, further research is required to precisely detail more these classes as well as their borderlines values.

The modeling equation that fits well the I_f-data evolution corresponding to the most inhibitory wood species (*Argania spinosa*) was found to be the logarithmic one with an R^2=0.99 (Figure 3). At WCR=20/200, this equation would give an I_f-value of 103.68%, exceeding then the 100% level. However with WCR=15/200, we obtain a value of 97.22% less than 100%. Based on this result, we can conclude that we should continue to use 15 grams-wood as a standard mass for estimating WC inhibition degree caused by integrating any wood species into mixtures. Also, the upper inhibition class could be I_f > 95% that would group all the most incompatible wood species that present a heat absorbing power such as the *Argania spinosa* case.

The hydration testing results, obtained based on the above standard WCR of 15/200, show that the I_f-values of the 12 studied wood species accurately reflect the positioning of their heat profile toward that of NC shown in Figure 4.

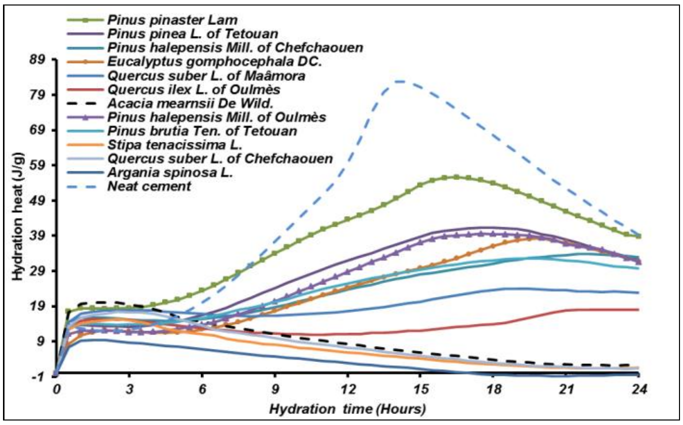

Figure 4: Hydration heat curves of neat cement and 12 Moroccan wood species.

The *Argania spinosa* heat curve continues to remain more contiguous to the X-axis with the highest value of I_f = 99,5%. Whereas, that of *Pinus pinaster* exhibit an extension toward the NC curve profile, recording the lowest value of I_f = 17,2 % .

Various WC inhibition estimating approaches are reported in Table 5 for the case of WCR=15/200 only, and including the newly proposed I_f-factor.

Table 5: Average wood-cement If-values observed for different types of biomass, as estimated by six different inhibition estimating methods for the case of wood-to-cement ratio of 15/200.

Scientific name	**Moroccan forest area**	**Designated wood-cement inhibition index (*) (%)**					
		Iw	**Ih**	**Ip**	**TR**	**Id**	**If**
Pinus pinaster Lam	*Kénitra*	4,9	31,1	38.1	131.1	18,3	17,2
Pinus pinea L.	*Tetouan*	13,3	39,9	50.0	139.9	28,1	31,4
Pinus halepensis Mill.	*Chefchaouen*	10,3	36,3	48.7	136.3	29,7	31,6
Pinus brutia Ten.	*Tetouan*	29,1	60,4	66.2	160.4	37,3	53,2
Eucalyptus gomphocephala DC.	*Maâmora*	23,8	56,1	59.4	156.1	43,4	36,9
P. halepensis Mill.	*Oulmès*	38,1	76,4	72.9	176.4	45,4	52,4
Quercus suber L.	*Chefchaouen*	39,1	63,5	72.4	163.5	55,2	66,2
Quercus ilex L.	*Oulmès*	45,7	70	77.3	170.0	70,2	71,2
Q. suber L.	*Maâmora*	30	-83	-16.6	17.0	90,5	89,7
Acacia mearnsii De Wild.	*Maâmora*	45,7	-85,2	-27.2	14.8	89,6	88,1
Stipa tenacissima L.	*Bouarfa*	27,4	-82	-13.7	18.0	91,6	91,8
Argania spinosa L.	*Admine*	22,7	-91	-10.3	9.0	95,7	99,5

(*) Iw: Index of Weatherwax and Tarkow (1964) ; Ih: Index of Hofstrand et al. (1984) ; Ip; Index of Pereira et al. (2006) ; TR: Index of Olorunnisola (2008) ; Id: Deduced deviation index of Hachmi et al. (2017) ; If: The newly proposed index.

Accordingly, the obtained WC I_f-values are dependent of the used method, which is clearly seen through the F-test results of ANOVA performed on data collected basically from all the five WCRs (Table 6).

Table 6: ANOVA between the newly proposed I_f-factor method, the I_d –value deduced from the Cs-modeling equation of Hachmi et al. (2017) and the four other inhibition indices (Weatherwax and Tarkow (1964), Hofstrand et al. (1984), Pereira et al. (2006), and Olorunnisola (2008)

Source of Variation	**Df**	**Squares**		**F**	**Pr > F**
		Sum	**Mean**		
Model	5	1160338,185	232067,637	132,090	0,000
Residual	1074	1886901,866	1756,892		
Total	1079	3047240,051			

The Duncan test reveals, at α = 0.05 level, the existence of four homogeneous subset groups (Table 7).

Table 7: Means of homogeneous subset groups based on Duncan test analysis.

Designated index*	**Sample size**	**Homogeneous subsets for alpha = 0.05**			
		1	**2**	**3**	**4**
Iw	180	17,3544			
Ih	180	24,9967			
Ip	180		35,9383		
Id	180			**45,7956**	
If	180			**49,1750**	
TR	180				117,4806
Sig.		,084	1,000	,445	1,000

* Iw: Index of Weatherwax and Tarkow (1964) ; Ih: Index of Hofstrand et al. (1984) ; Ip; Index of Pereira et al. (2006) ; TR: Index of Olorunnisola (2008) ; Id: Deduced index of Hachmi et al. (2017) ; If: The newly proposed index.

The first group included two indices: Iw of Weatherwax and Tarkow (1964) and Ih of Hofstrand et al. (1984) with a mean value of 17 and 25 %, respectively. The third group included the Id-index and the newly proposed I_f-factor with mean values of 46 and 49%, respectively (Table 7). These two latter indices are once again not significantly different from each other. This means that from nowadays the researchers in WC research field have choice to use either the I_f-factor, estimated through heat curve characteristics, or the compatibility index expressed as a temperature-curve similarity coefficient (C_S) that let to deduce the inhibitory index I_d-value based on the principle that the summation of the absolute values of the two latter indices equals to 1 ($I_d + C_S = 1$). The second and fourth group correspond to the Ip-index of Pereira et al. (2006) with a mean value of 36% and TR-index of Olorunnisola (2008) with a mean value of 117%, respectively.

CONCLUSION

A new alternative factor to estimate WC hydration inhibition was developed based on equivalent heats difference concept. This difference, which is weighted to the NC control heat, involves in first step three heat quantities (Q in Joules/gram): Q1 of the first reaction, Q2 of the second reaction and Qcr the residual cooling heat recorded at 24h hydration limit. The obtained ratio is adjusted by a coefficient that includes the WCR parameter and the time to reach maximum heat released by the second cement exothermic reaction. The results of the ANOVA, performed on six inhibition estimating methods applied on five WCRs, reveal that there is no significant difference between the newly proposed I_f-factor and the I_d-index deduced from the previously developed modeling C_S-index equation. But both of these two indices are significantly different from the other four remaining compared indices. As result, using either I_f-factor or C_S-index (I_d=1-C_S) would lead to the same result. Another finding is that the WCR parameter has a significant effect on I_f-value, resulting in defining different evolution trends: linear, logarithmic, polynomial, and power equation shape, depending on wood species types. These different evolution trends may seriously constitute a basis for identifying the WC inhibition classes. Also, to avoid getting I_f-values > 100% limit, we should continue to use 15 grams of wood as a standard mass to estimate WC inhibition.

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