

EFFECTS OF COTTON STALKS PARTICLES ON THE HYDRATION AND MECHANICAL PROPERTIES OF PORTLAND CEMENT

E. Abdel Ghafar¹, F. Heider¹, M. Shehata¹, M. Morsy²

ABSTRACT

The purpose of this research is to study the effect of cotton stalks fiber on the hydration and mechanical properties of Portland cement. Cotton stalks particles (20 – 40 mesh) was mixed with Portland cement 32.5 R at 7.5 % by weight. Three treatments were done to determine the impact on cotton stalks cement compatibility; water extraction, 1% NaOH extraction, and chemical additives based on 3% of cement weight. CaCl₂, MgCl₂, FeCl₃ and NaOH were used as a chemical additives.

Based on the results obtained from hydration and compressive strength under untreated conditions, cotton stalks cement mixture had low level of compatibility. Treating cotton stalks with hot water or NaOH solution improving the compatibility between cotton stalks and cement but the inhibitory effect remain high. Generally, adding 3% CaCl₂ to hot water treated cotton stalks particles overcome this problem and the compressive strength of the composites was also improved.

Keywords: cotton stalks; Cement; Composite; Hydration; Compatibility; compressive strength.

INTRODUCTION

Cement composites out of wooden particles have been produced from a number of agro-forestry material including sawdust, construction waste, bagasse, coffee husk, maize husk, and ratten furniture waste among others (Kasai et al 1998, Olorunnisola and Adefisan 2002, Ajayi 2002).

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Cement wood composites are well established in market place. This is because, for some application, like prefabricated construction, wood cement composites, or in more general sense, composites made of cement and lignocelluloses materials, have advantages over most common wooden composites including organic resin -bounded panels. Compared with these, wood-cement composites have better dimensional stability, better resistance to biodeterioration and fire, have no formaldehyde emission originating from the binder, and can be used as means of recycling wood residues (Pererira et al. 2006). However, when particles from many wooden materials come in contact with cement slurry, their organic compounds such as soluble sugars, hemicelluloses, lignin, fatty acids, terpenes, etc. tend to retard or inhibit cement hydration and bound formation between cement and wooden particles (Biblis & Lo 1968, Zhengtian & Moslemi 1986, Miller and Moslemi 1991, and Alberto et al.2000). Several measures of minimizing the effect of water solubility have been devised, including prolonged storage of the wood materials, hot or cold water extraction of soluble sugars and the use of chemical accelerators, namely dilute sodium hydroxide (NaOH), sodium silicate (Na_2SiO_3), calcium chloride (CaCl_2) and aluminum sulphate ($\text{Al}_2(\text{SO}_4)_3$) among others (Lee & Hong 1986 and Badejo 1989). Using of CaCl_2 with a dosage less than 4% by weight of cement tends to accelerate the hydration of wood cement mixture, thereby enhancing the wood cement bound and the mechanical properties of the composites (Biblis & Lo 1968, Zhentian & Moslime 1986, Moslime & Pfister 1987, Wie et al 2000, and Olorunnisola & Adefisan 2002). The compatibility of lignocellulose substances can be assessed by thermal and mechanical methods. Cement hardening is exothermic, and a plot of temperature versus time can be used to assess compatibility. Several parameters can be obtained from every plot such as: T (maximum temperature attained), t (time to T) and S (maximum slope during cement setting phase). Compatible lignocelluloses would have T, t, and S that are similar to neat cement paste. Therefore, these factors taken alone, or as basis for calculation of indices, have been used to indicate the degree of compatibility between cement and wood species (Sandermann & Kohler 1964, and Weatherwax & Tarkow 1964). However, In order to obtain

experimental data that may correlate better or more consistently with the properties of the final product made on a larger scale, Lee and Hong (1986) suggested a simple compression test of cylindrical samples, and Wei et al. (2000) measured modulus of rupture (MOR) and internal bond (IB) in panels, for the same purpose.

The present work aimed to investigate the effect of cotton stalks treatments (hot water treatment and 1% NaOH) and chemical additives (CaCl₂, MgCl₃ and FeCl₃) on the compatibility of cotton stalks with cement.

MATERIAL AND METHODS

Cotton stalks collection and preparation

Cotton stalks collected from the experimental station of Faculty of agriculture, Alexandria University, Alexandria, Egypt. Cotton stalks particles were prepared by milling cotton stalks by hammer mill then sieved to take the particle between 20~40 mesh (Figure 1).

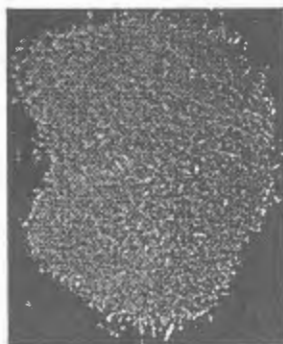


Figure 1. Cotton stalks particles.

Specifications of cement, additives and water

Fresh Portland cement was used (class strength 32.5 R grade, graded in accordance with BS EN 197-1:2000). Cement was stored in air-tight containers. CaCl₂, MgCl₃, FeCl₃ and NaOH powders (technical grade) was used as chemical additives. Distilled water at room temperature (20 ± 2 oC) was used.

Treatment measures on cotton stalks particles

Treatment measures employed on the cotton stalks particles are as shown in Table 1. They included extraction and Chemical additives.

Table 1.

Treatments measures performed on rattan particles

Treatment	Description
Controuls	<ul style="list-style-type: none"> • Neat Cement • Untreated cotton stalks +Cement
Measure 1	<ul style="list-style-type: none"> • Hot water treated cotton stalks + Cement • NaOH treated cotton stalks + Cement
Measure 2	<ul style="list-style-type: none"> • Untreated cotton stalks +Cement + 3% CaCl₂ • Untreated cotton stalks +Cement + 3% MgCl₂ • Untreated cotton stalks +Cement + 3% FeCl₃ • Untreated cotton stalks +Cement + 3% NaOH
Measure 3	<ul style="list-style-type: none"> • Hot water treated cotton stalks + Cement +3% CaCl₂ • NaOH treated cotton stalks + Cement + 3% CaCl₂

To remove at least a portion of cotton stalks extracts and sugars we initiated two major treatments as follows:

Hot water treatment

The amount of cotton stalks particles were extracted in boiling water for 3 hours while changing water every one hours. After that cotton stalks particles were collected after sieving through 200 mesh screen, then washed twice with hot distilled water and dried for 24 hours in an oven at 105 ± 5 °C

Sodium hydroxide treatment (NaOH)

The amount of cotton stalks particles were soaked in one percent of sodium hydroxide solution for 24 hours. The cotton stalks milled were collected, washed and dried according to the method outlined above.

Chemical additives

CaCl₂, MgCl₂, FeCl₃ and NaOH were used as a chemical additive to enhance cement setting. Three percent (from cement weight) of each chemical additive was dissolved in distilled water added to the dry cotton stalks cement mixture.

Cotton stalks cement mixture preparation

To discover the retardation effect of cotton stalks on Portland cement hydration, untreated and treated cotton stalks particles 20~40 mesh with or without chemical additive was mixed with cement at 7.5% from the cement weight. The amount of water for the cement cotton stalks mixture was calculated by the following equation:

$$WC \text{ (the amount of water, g)} = 0.25 \text{ (mass of cement, g)} + 2.7 \text{ (mass of cotton stalks particals, g)}$$

A homogenous mixture is given by mixing cement with cotton stalks by hand, adding water and remixing again for 2 minutes.

Expermintal tests

Two testes were used to investigate the effect of adding cotton stalks particels on cement hydration, as follows:

Hydration test

The hydration test method used was the same as described by Okino et al. (2004), Olorunnisola et al. (2008), Moslemi and Lim (1984) and Weatherwax and Tarkow (1964) and Hachimi et al. (1990). The hydration tests were conducted in sealed thermally insulated containers (Dewar flasks) in which T-type thermocouples, connected to a multipoint data recorder, were inserted. The exothermic phenomenon of the cotton stalks cement water mixture was observed and plotted against time. Temperatures were recorded at 5 min intervals. The ambient temperature is ranging from 20 to 22 °C throughout the experiment. The inhibitory index (I) was calculated using following equations:

$$I, \% = 100. [((T-T)/T) * ((t-t)/t) * ((S-S)/S)]$$

Where:

T and T: are the maximum hydration temperatures of neat cement and mixture respectively, measured by(°C).

t and $t\backslash$ are the required time to reach maximum hydration temperature
 : of neat cement and mixture respectively, measured by (h).
 S and are the maximum slopes of curves for neat cement and mixture
 $S\backslash$: respectively, measured by ($^{\circ}\text{C}/\text{h}$).

The compatibility levels of the cotton stalks samples with Portland cement were assessed using the three schemes shown in Table 2 as proposed by Hofstrand et al.(1984), Sandermann & Kohler(1964), and Hächmi & Moslemi (1989). The three schemes were developed based on the assumption that hydrated neat cement and wood–cement–water mixtures exhibit the same hydration behavior except that the hydration rate of wood–cement mixtures tends to be generally lower. Hence compatibility of wood with cement is assessed by comparing hydration rates (i.e., setting time, the maximum hydration temperature, and inhibitory index) of wood –cement–water mixtures with that of neat cement. The compatibility levels indicated in the three schemes were set based on the assumptions that the setting (time to reach maximum hydration temperature) of neat cement typically takes about 8 h for Scheme 1; the maximum hydration temperature tends to exceed 60 $^{\circ}\text{C}$ for Scheme2; and that the inhibitory index is a good indicator of cement hydration behavior for Scheme 3 (Okino et al. (2004), Sandermann & Kohler (1964), Hachmi & Moslemi (1989).

Compressive test:

The previous cement cotton stalks mixture were formed in a cylinder mold (50 mm diameter / 100 mm length) and covered with plastic sheet at room temperature. After 1 days the specimen were removed from the molds and wrapped in a plastic bags for further curing ages at room temperature. Three periods were utilized as a curing ages, namely 7, 14, and 28 days. Three specimens were assigned for each curing age. Compressive strength (CS) for all specimens was carried out according to the ASTM C – 39, 1984.

Table 2. Methods employed to assess aggregate/cement compatibility

S. No.	Parameter	Classification Grade
1	Setting time (t)	<ul style="list-style-type: none"> • Suitable ($t \leq 15$ h) • Intermediately suitable ($15 \text{ h} > t < 20$ h) • Unsuitable ($t > 20$)
2	Maximum hydration temperature (T)	<ul style="list-style-type: none"> • Suitable ($T > 60$ °C) • Intermediately suitable ($50 > T < 60$ °C) • Unsuitable ($T < 50$ °C)
3	Inhibitory index (I)	<ul style="list-style-type: none"> • Low inhibition ($I < 10$) • Moderate inhibition ($I = 10 \sim 50$) • High inhibition ($I = 50 \sim 100$) • Extreme inhibition ($I > 100$)

RESULTS AND DISCUSSION

Effect of Cotton stalks treatment on cement hydration

Figure 2 shows the hydration curve of neat cement and cotton stalks cement mixtures. From Figure 2, it appears that the second peak of hydration of C_3S totally disappear by additional of untreated cotton stalks particle. That's meaning, cotton stalks had a very high effect on delaying the hydration reaction of cement. The correspondent parameter-values of the hydration test untreated and treated cotton stalks cement mixture and its compatibility classification are listed in Table 3.

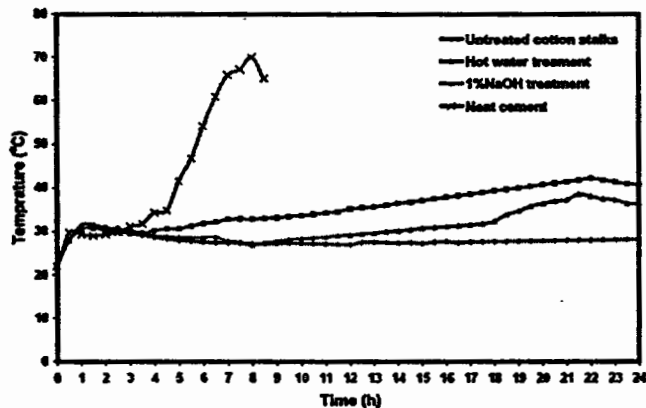


Figure 2. Hydration curve for treated cotton stalks cement water system. The treatment included removal of soluble extracts by hot water or NaOH solution. A comparison is made with neat cement and untreated data.

Table 3. Influence of cotton stalks treatment on hydration of cement

Treatment	Parameters			Compatibility level		
	t(h)	T (°C)	I (%)	S.1	S.2	S.3
Neat cement	8	70	-	-	-	-
Untreated cotton stalks + Cement	36	30	181	U	U	EI
Water treated cotton stalks + Cement	21	40	58.93	U	U	HI
NaOH treated cotton stalks + Cement	21.5	38	60.51	U	U	HI

S.1= compatibility level based on setting time.

S.2= compatibility level based on maximum hydration temperature.

S.3= compatibility level based on area ratio.

S= suitable; IS= intermediately suitable; U= unsuitable; LI= low inhibition; MI=moderate inhibition; HI= high inhibition; EI= extreme inhibition.

Statistical analysis of the result shows significant differences among T, t and I values of the treated and untreated cotton stalks cement mixtures.

The results in Table 3 indicates that the untreated cotton stalks cement mixture completely failed to set over 72 h test period. However, when cotton stalks treated, the hardening of the mixture took place, T values increased significantly. While, t and I values decreased significantly. Furthermore, hot water performed better in reducing t value and in elevating T value. Based on I value, the corresponding grade for untreated cotton stalks cement mixture is „ Extreme Inhibition “. While, treated cotton stalks can characterized with the grade „ High Inhibition“.

Effect of chemical additives on hydration of cotton stalks cement mixture.

The influence of chemical additives on cotton stalks- cement- water systems involved to two phases. The first phase was designed to determine influence of chemical additives on untreated cotton stalks cement mixture. While, the second phase was designed to determine the influence of the best chemical additives in the first phase on treated cotton stalks cement mixture.

Untreated Cotton stalks

In a series of preliminary tests, 3% solution concentration of CaCl_2 , MgCl_2 , FeCl_3 and NaOH were used in combination with cotton stalks cement mixture. The proportion of cotton stalks, cement and water was that indicates earlier in this paper. Figure 3 shows the effect of the used chemical additives on the hydration curve of untreated cotton stalks cement mixture. The results of the preliminary tests are shown in Table 4.

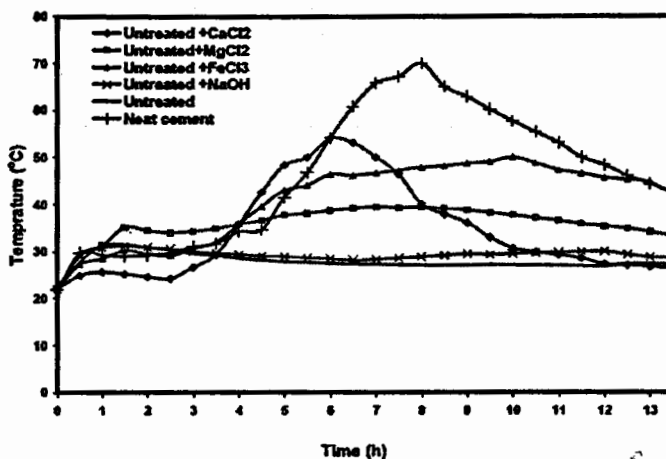


Figure 3. The effect of chemical additives on the hydration curve of untreated cotton stalks cement water system. A comparison is made with neat cement and untreated data.

Table 4. Influence of chemical additive on hydration of cotton stalks cement mixture

Treatment	Parameters			Compatibility level		
	t(h)	T(°C)	I(%)	S.1	S.2	S.3
Untreated cotton stalks + Cement	72	30	421	U	U	EI
Untreated cotton stalks + Cement + CaCl_2	6	54.1	8.12	S	IS	LI
Untreated cotton stalks + Cement + MgCl_2	8	39.5	.0	S	U	LI
Untreated cotton stalks + Cement + FeCl_3	10	50	3.5	S	IS	LI
Untreated cotton stalks + Cement + NaOH	24	29.8	105	U	U	EI

S.1= compatibility level based on setting time.

S.2= compatibility level based on maximum hydration temperature.

S.3= compatibility level based on area ratio.

S= suitable; IS= intermediately suitable; U= unsuitable; LI= low inhibition; MI= moderate inhibition; HI= high inhibition; EI= extreme inhibition.

The analysis of these results shows that the addition of CaCl_2 , MgCl_2 and FeCl_3 reduces the t and I values significantly. It is also interesting to note that T was increased significantly. The addition of NaOH to untreated cotton stalks cement mixture did not produce any noticeable effect in the exothermic reaction of the mixture (Figure 3).

Based on I value, the corresponding grade for untreated cotton stalks cement mixture with CaCl_2 , MgCl_2 and FeCl_3 as a chemical additives are „Low inhibition“. While, untreated cotton stalks cement mixture with NaOH as a chemical additive characterized with the grade of „High Inhibition“

The results also shows that, among the additives included, CaCl_2 appear to bring about better response in improving the hydration reaction and strength development. Therefore, CaCl_2 was selected for further experimentation.

Effect of CaCl_2 additive on hydration of treated cotton stalks cement mixture.

In this test series, we intended to determine the effect of CaCl_2 on the exothermic behavior of cement when combined with treated cotton stalks with hot water or with 1% NaOH, as described earlier. Results of these testes are presented in Table 5 and shown in Figure 4. An analysis of these data indicates that the addition of CaCl_2 into treated cotton stalks cement mixture produced a significant reduction in the t and I values as well as an increase in the T value. The addition of CaCl_2 to untreated cotton stalks cement mixture resulted in a reduction of the average t values from 72 h to 6 h, with an increase in the average T values from 30 to 54°C. However, the addition of CaCl_2 to hot water treated cotton stalks mixture results in a reduction of t value from 22 h to 5 h with an increase in T value of 23°C (42°C to 65°C). Similar results were obtained by adding the CaCl_2 to NaOH treated cotton stalks cement mixture (t value decreased from 21.5h to 5.5h and T value increased from 38 to 60°C).

Based on I value, The corresponding grade for treated cotton stalks cement mixture with CaCl_2 as a chemical additives are „Low inhibition“.

Table 5. Influence of CaCl_2 on hydration of treated cotton stalks cement mixture

Treatment	Parameters			Compatibility level		
	t(h)	T (°C)	I (%)	S.1	S.2	S.3
Untreated cotton stalks + Cement + CaCl_2	6	54.1	8.12	S	IS	LI
Hot water treated cotton stalks + Cement + CaCl_2	5	65.6	1.3	S	S	LI
NaOH treated cotton stalks + Cement + CaCl_2	5.5	60	0.79	S	S	LI

S.1= compatibility level based on setting time.

S.2= compatibility level based on maximum hydration temperature.

S.3= compatibility level based on area ratio.

S= suitable; IS= Intermediately suitable; U= unsuitable; LI= Low inhibition; MI= Moderate inhibition; HI= High inhibition; EI= Extreme inhibition

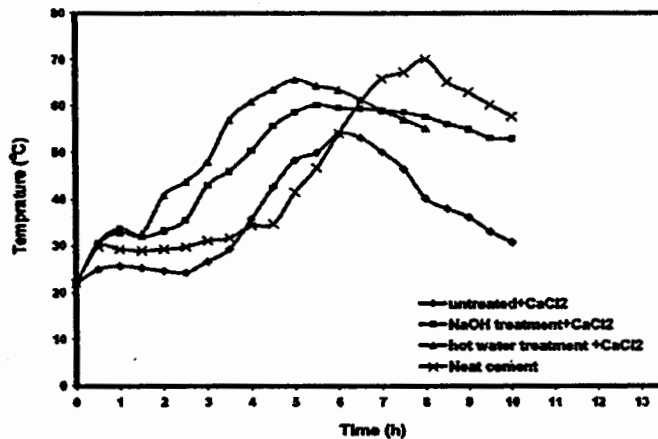


Figure 4. The effect of CaCl_2 on the hydration curve of treated cotton stalks cement water system.

Compressive strength (CS)

Figure 5 shows the effect of treatments and chemical additives on the strength development of cotton stalks cement mixture at 7, 14 and 28 days. An analysis of these data indicates that the addition of cotton stalks particles to cement paste with 7.5 % (by weight) produced a significant reduction in the compressive strength (value decreased from 31.2 MPa to 1.8 MPa at 28 day). By treating cotton stalks with hot water or with NaOH solution a significant improvement in the strength was observed. Compressive strength increased from 1.8 MPa (for untreated cotton stalks cement mixture) to 13.4 MPa and 8.9 MPa (for hot water treated and NaOH treated cotton stalks cement mixture, respectively).

However, the addition of CaCl_2 , MgCl_2 , and FeCl_3 increasing the strength significantly. Compressive strength increased from 1.8 MPa for untreated cotton stalks cement mixture to 12.4, 9.1 and 11.6 MPa for untreated cotton stalks cement mixture with CaCl_2 , MgCl_2 , and FeCl_3 , respectively. While, the addition of NaOH to untreated cotton stalks cement mixture did not produce any noticeable effect on the strength development. Furthermore, the addition of CaCl_2 to hot water or NaOH treated cotton stalks cement mixture resulting in further increases the strength development. Compressive strength increased from 12.4 MPa for untreated cotton stalks cement mixture with CaCl_2 to 18.7 MPa and 26 MPa for NaOH and hot water treated cotton stalks cement mixture with CaCl_2 .

Hydration data and compressive strength relationship

Correlating the properties of the foregoing cotton stalks cement composites, and based on the value of R^2 , it is obvious that, there were good correlation between the compressive strength and the hydration data (Figures 5, 6 and 7). This indicates that the change in the hydration properties of cotton stalks cement composite by treatments, significantly affects the strength properties.

CONCLUSION

From all the foregoing results, the following conclusions can be drawn:

1. From the hydration data and compressive strength, untreated cotton stalks was classified as unsuitable to addition with cement.
2. The difference between the hydration data between the treated and untreated cotton stalks cement mixture was significant, also compressive strength was significantly increased under treated condition.
3. The addition of CaCl_2 , MgCl_2 and FeCl_3 to untreated cotton stalks cement mixture improving the hydration process and the strength development, but the effect of CaCl_2 was better than the effect of MgCl_2 and FeCl_3 .
4. The addition of CaCl_2 to hot water treated cotton stalks cement mixture gave the highest increase in compressive strength and best hydration data.

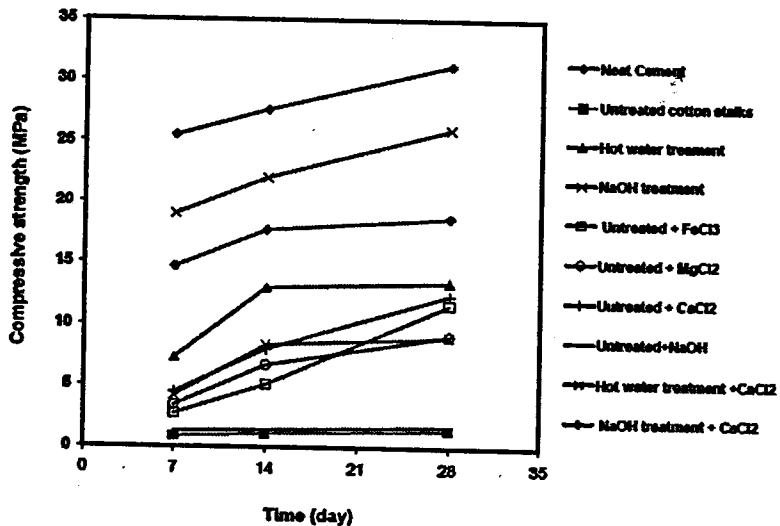


Figure 5. effect of treatments and chemical additives on the compressive strength of cotton stalks cement water system

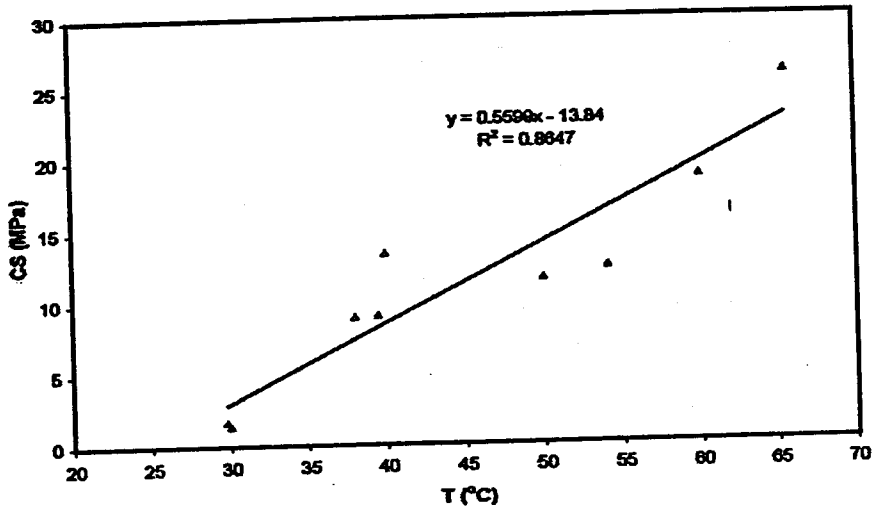


Figure 5. Correlation between the maximum hydration temperature and compressive strength

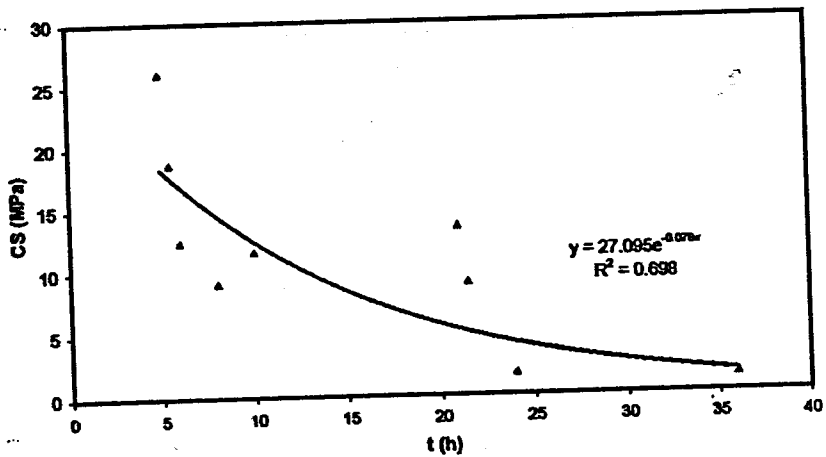


Figure 6. Correlation between time to reach the maximum hydration temperature (setting time) and compressive strength

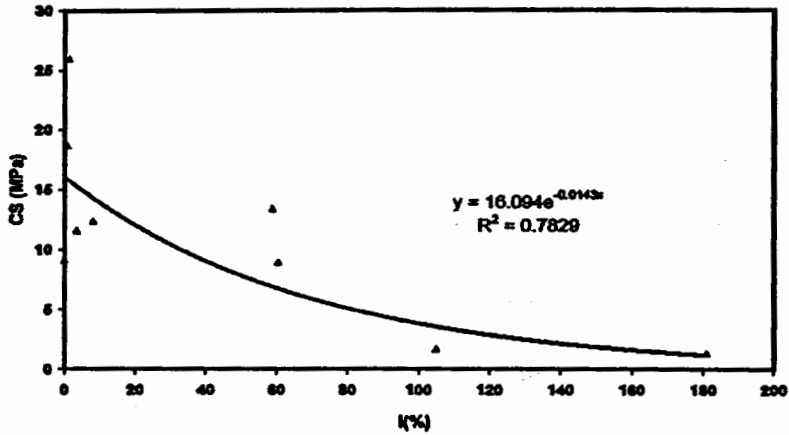


Figure 7. Correlation between inhibitory index and compressive strength

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الملخص العربي**تأثير إضافة حبيبات حطب القطن على تفاعل الإماهة والخواص الميكانيكية للأسمنت البورتلاندى**

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الهدف الرئيسى لهذا البحث هو دراسة تأثير إضافة حطب القطن على تفاعل الإماهة والخواص الميكانيكية للأسمنت البورتلاندى. تم خلط حبيبات حطب القطن (٢٠ إلى ٤٠ مش) بالأسمنت البورتلاندى العادى (R 32.5) بمحتوى ٧.٥% من وزن الأسمنت.

لدراسة تأثير المعاملات على توافق حطب القطن مع الأسمنت تم استخدام ثلاثة معاملات رئيسية وهى:

- ١- الاستخلاص بالماء الساخن
- ٢- الاستخلاص بمحلول هيدروكسيد الصوديوم ١%
- ٣- تأثير إضافة كل من كلوريد الكالسيوم - كلوريد الماغنسيوم - كلوريد الحديدك - هيدروكسيد الصوديوم كمسرعات شك للأسمنت بنسبة ٣% من وزن الأسمنت.

طبقا للنتائج المتحصل عليها من تفاعل الإماهة وإجهاد الضغط لخليط حطب القطن بدون معاملة. والأسمنت فإن مقدار التوافق بين الحطب والأسمنت منخفض. من جهة أخرى إن معاملة حطب القطن بالماء الساخن أو هيدروكسيد الصوديوم أدى إلى تحسين مقدار التوافق بين حطب القطن والأسمنت ولكن معامل تثبيط التفاعل ظل مرتفعا وعلى وجه العموم فإن إضافة ٣% من وزن الأسمنت كلوريد كالسيوم لخليط حطب القطن (المعامل وغير المعامل) والأسمنت أدى إلى زيادة مقدار التوافق بين حطب القطن والأسمنت بالإضافة إلى الزيادة المعنوية فى إجهاد الضغط.

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