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Suitability of Vine (*Vitis vinifera* L.) Prunings for Wood-Cement Industry

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Abstract: The aim of the current investigation was to study the suitability of pruning of vine (*Vitis vinifera* L.) as a lignocellulosic material for the production of wood-cement composite panels, enhance their compatibility with cement using various pretreatments and chemical additives. Hydration tests of net cement and vine-cement mixture were carried out using a 2-litre Dewar flask. Suitability was determined by inhibitory index (I) and compatibility factor (C_A), which were calculated from the hydration data. Under untreated condition, vine pruning particles were incompatible with cement and were classified as "unsuitable" for making wood-cement boards. Using of cold or hot water extraction for those materials enhanced their compatibility with cement. However, it can be reclassified as 'suitable under limited conditions' for making wood-cement boards only by using hot water extraction. Addition of 3% $CaCl_2$ to untreated vine particles resulted in its reclassification as 'suitable under limited conditions' (T_{max} value was 39.27°C and C_A value was 69.37%). These results suggested that vine grape prunings can be used to produce wood-cement panels after extraction by hot water or addition of 3% $CaCl_2$ as an accelerator.

Key words: Chemical analysis · Compatibility · Hydration test · Portland cement · Wood-cement panels · Vine

INTRODUCTION

Saudi Arabia like many countries in arid and semi-arid zones is poor in natural forest [1]. Increasing demand of forest resources for wood fuel and wood industries leads to continuous effort of finding an alternative raw material to forest wood [2]. Researches to find this alternative material have been conducted by both industry and research institutions. Agricultural residues are renewable resources that can be utilized as raw materials for wood industry [3,4]. A wide variety of agricultural residues and non-woody materials, such as midribs of date palm [5], wheat straw [1], rattan [4], arhar stalks [6], bamboo [7] and coconut shell of babacu [3] have been studied. Fortunately, Saudi Arabia has relatively large quantities of lignocellulosic materials, including the vine prunings available in the form of agricultural residues. A large vine population in Saudi Arabia sheds huge quantity of residues annually as a result of seasonal pruning, which is an essentially agricultural practice. It can be speculated that about 3-8 kg of biomass are wasted annually from seasonal pruning of the vine grape depending on the variety, soil type, region and agricultural treatments. The average pruning yield is about 5 tons per hectare [8]. In the developed countries these residues are used to produce valuable wood-based panels such as particleboard and medium density fiberboard (MDF), although in most of the developing world these are simply burnt to ashes [5].

The grape vine (*Vitis vinifera* L.) is an important economic fruit crop in many countries in the world. In Saudi Arabia, its orchards have recently increased to 11675 hectares with an average production of 144430 tons [9]. Some of the vine prunings are used as fuel but most of it remains unused on the field. Utilization of this waste in the production of wood-cement panels would be welcomed by the cultivators.

Wood-cement particleboards (WCP) are used as reinforcement materials, bonded and held together by an inorganic binder such as ordinary Portland cement [10]. Using varying methods and techniques, lignocellulosic materials from different countries have been screened, tested and classified according to their suitability for wood-cement panel production [4, 11-15].

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Unlike the resin-bonded particleboards, WCP show excellent sound insulation, high resistance to water and termites and excellent long-term weatherability in outdoor conditions [16, 17]. These advantages confirm their potential application for replacing the traditional building materials and conventional wood composites as siding, roofing, wall and flooring parts, support tables and noise absorbing partitions [18].

With all the advantages of WCP, however, the compatibility of some lignocellulosic materials with cement is limited, which may inhibit cement setting to some extent and limit WCP development [13, 19, 20]. The water-soluble materials in wood have the greatest inhibitory effect [10, 14, 16, 21, 22]. Extraction of wood by hot or cold water and/or addition of chemical additives to wood-cement mixtures are important in improving their compatibility [16, 23].

WCPs are generally made from wood species, which have been tested and proved suitable for the purpose [24]. The compatibility of wood-cement mixture is measured normally by the method developed by Sandermann and Kohler [21], which is reliable and simple. This compatibility is classified on the basis of the extent of retarding the cement hydration.

Studies conducted in Saudi Arabia to produce wood-cement particleboard and to evaluate the compatibility of agricultural residues for WCP manufacturing are limited. Therefore, the objective of the current study was to evaluate the suitability of vine prunings to replace wood as raw materials for manufacturing wood-cement products that have a high economical value. Results of this work were compared with those obtained from the mixture of cement and Scots pine (*Pinus sylvestris*), a species considered suitable for the manufacture of wood-cement composites [15].

MATERIALS AND METHODS

Raw Material: The lignocellulosic material used for the current study was the pruning residue of vine (*Vitis vinifera* L.) collected in December 2010 from the seasonal pruning process of Thompson seedless vine grapes planted at the Agricultural Experimental Station near Derab, 50 km south of Riyadh in Saudi Arabia. For comparison wastes of the European redwood (*Pinus sylvestris* L.) were collected from wood stores in Riyadh city. These materials were air-dried and reduced to small pieces (about 2-5 cm long) in order to facilitate grinding into meal. These were later put in a prototype hammer mill and screened. Wood meal passing through a 20-mesh screen and retained on a 40-mesh screen was used for hydration test, while those meals that pass through a 40-mesh screen and retained on a 60-mesh screen were used for chemical analysis.

Commercial ordinary Portland cement (Type 1), meeting ASTM C150-84 specification and manufactured by Al-Yammama Cement Company, Riyadh was used as a binder.

Chemical Additives: Calcium chloride dehydrate ($CaCl_2.2H_2O$) and magnesium chloride ($MgCl_2$) at 1 and 3% of cement weight were used as chemical additives. These were dissolved in distilled water at least 15 minute before adding to the untreated wood-cement mixture.

Cold and Hot Water Treatments: Oven-dried wood meal (about 30 g, 20-40 mesh) was soaked in cold distilled water or extracted in boiling distilled water for 48- hours or 3 hours according to Moslemi *et al*. [10] with some modification. The pH of the extracts was measured with a pH meter by using distilled water (pH=6.5).

Seven treatments, namely, wood+cement (untreated, UNTRT), wood extracted by cold water+cement (CWE), wood extracted by hot water+ cement (HWE), UNTRT + 1%$CaCl_2$, UNTRT+3%$CaCl_2$, UNTRT+1%$MgCl_2$ and UNTRT+ 3%$MgCl_2$ were used in this study.

Chemical Analysis of Lignocellulosic Materials: The contents of total extractives, cellulose, hemicellulose and ash for vine grape and European redwood were determined according to the standard methods as follows:

Extractives Content Determination: The extractive content of wood was determined according to the ASTM, D1105-84 [24]. Samples of air-dry wood were chipped and ground to pass through 40-mesh screen and retained on a 60-mesh screen. Air-dried wood meal was extracted in a Soxhlet apparatus with ethanol-benzene mixture (in the ratio of 1:2 by volume, respectively) for four hours, followed by extraction with 95% ethanol for four hours and finally extracted with hot distilled water for four hours with chancing water every one hour. The percentage of extractives was calculated based on the oven-dry weight of wood samples.

Cellulose Content Determination: Cellulose content was determined by the treatment of extractive-free wood meal with nitric acid and sodium hydroxide: one gram of extractive-free wood meal was treated with 20 ml of a solution of nitric acid 3% in flask and was boiled for

30 min. The solution was filtered in a crucible. The residue was treated with 25 ml of a solution of sodium hydroxide 3% and was boiled for 30 min. The residue was filtered, washed with warm water to neutral filtrate, dried and weighed. The cellulose content was calculated as percentage of residues based on oven dry wood meal weight [25].

Hemicelluloses Content Determination: Hemicellulose content of buttonwood samples was determined by the treatment of extractive-free wood meal (about 1.5 g) with 100 ml sulfuric acid (2%) and boiled for 1 hour under a reflex condenser and filtrated in a crucible. After that the residue was washed with 500 ml of hot distilled water to free of acid and contents were dried in an oven at 105± 2°C, cooled in a desiccator and weighed [26]. Then, the hemicellulose content was calculated based on the oven-dry weight of the spacemen.

Ash Content Determination: Ash content of wood was determined according to the Chemical Analysis and Testing Task Laboratory Analytical Procedure “NREL” [27]. Approximately one gram of oven-dry sample was placed into the crucible. The sample in an uncovered crucible was heated gradually, then ignited at 575±25°C in muffle furnace for a minimum of three hours, or until all the carbon is eliminated. Ash content was calculated as a percentage of residues based on the oven-dry wood meal weight.

Hydration Procedure: The hydration of the net cement and wood-cement mixtures was carried out according to Hofstrand *et al*. [11]. 200 grams of cement and 15-grams of oven-dried milled wood (20-40 mesh) were mixed and kneaded with 90.5 ml of distilled water for about two minutes. The mixture was then placed into a two liter Dewar flask. The thermocouple wire (Type T) was connected to a data-logger where the temperature was measured at 15-minute intervals for 24 h. The time and temperature readings were plotted to obtain the exothermic hydration curve. The hydration data were used to calculate compatibility factor, C_A, [28] and inhibitory index, I, [11]. The 24-h limit was chosen for practical reasons in order to limit the hydration test duration [29].

Statistical Analysis: The experimental design was the split-plot design [30]. The main plot was for the lignocellulosic materials, while the treatments (pretreatments and chemical additives) were used as sub-plot, with three replicate. The results were analyzed using Statistical Analyses System [31]. Least significant difference at 95% level of confidence ($LSD_{0.05}$) method was used to detect the differences between means.

RESULTS AND DISCUSSION

The results (T_{max} and t_{max}) obtained from the hydration test of vine-cement and European redwood-cement mixtures are presented in Table (1).

Suitability of the Untreated Vine Particles for WCP Industry: The statistical analysis of the data revealed that in all the hydration parameters of wood-cement mixtures the vine grape significantly differed from the European redwood. Figure (1) presents the exothermic hydration curves of the untreated lignocellulosic materials with cement. It shows that each material reacted differently when mixed with cement. The extents of suppression temperature and t_{max} were measured to determine the retarding effect of the wood and non-woody lignocellulosic materials on cement setting. The wood, which caused greater temperature depression, is likely to be less suitable for WCP manufacture.

Compared with net cement, the untreated lignocellulosic materials used in this study depressed the temperature rise and increased the time to reach the maximum temperature during the cement setting

Table 1: Hydration data for untreated vine grape-cement mixture in comparison with European redwood-cement mixture and net cement

Mixture	T_{max} (°C)	t_{max} (hrs)	ΔT (°C)	R (°C/hr)	C_A (%)	I (%)
Vine particles-cement	40.33^B	24^A	16.33^B	0.68^B	24.96^B	125.93^A
European redwood-cement	52.50^A	9.93^B	27.80^A	2.80^A	78.49^A	13.24^B
Net cement	83.60	6.28	59.20	9.43	100.0	0

Means with the same letters in the same column are not significantly different at 5% level of probability according to LSD test

T_{max}:maximum temperature. t_{max}: time to reach T_{max}.

ΔT: rise in temperature above the ambient. R: hydration rate.

C_A: compatibility factor. I: inhibitory index.

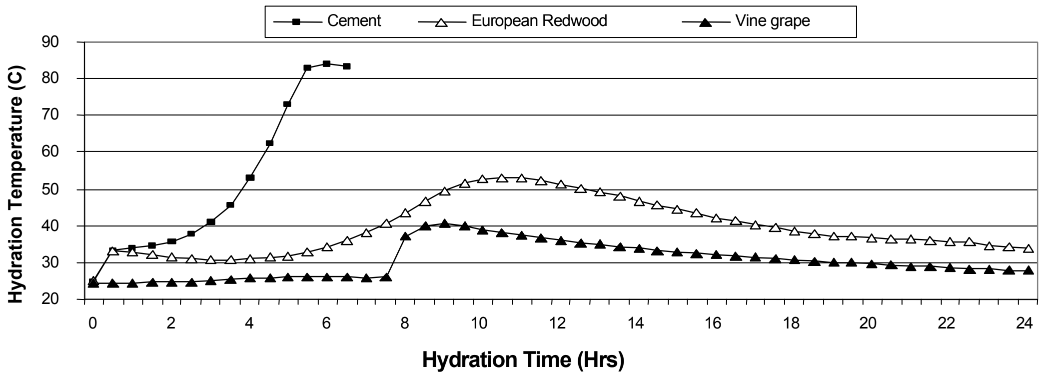

Fig. 1: Exothermic hydration curves of untreated vine particles-cement and European redwood-cement mixtures without chemical additives comparing with net cement.

Table 2: Chemical analysis of vine particles and European redwood used for wood-cement mixtures

Lignocellulosic Materials	% of total material weight				Hot water		Cold water	
	Total extractives	Cellulose	Hem-celllulose	Ash	Solubility (%)	pH	Solubility (%)	pH
Vine particles	18.85^{A}	38.14^{B}	33.76^{A}	4.421^{A}	18.77^{A}	4.42^{B}	13.65^{A}	4.64^{B}
European redwood	6.65^{B}	53.47^{A}	16.99^{B}	0.30^{B}	6.03^{B}	5.70^{A}	3.13^{B}	5.87^{A}

Means with the same letters in the same column are not significantly different at 5% level of probability according to LSD test

process (Table 1 and Fig. 1). European redwood (*Pinus sylvestris*) gave a higher T_{max} (52.50°C) and a lower t_{max} (9.93 hrs) in comparison to vine grape i.e. T_{max} (40.33°C) and t_{max} (24 hrs). According to these values, the former had a higher C_A (78.49%) and lower I (13.24%) values, while the latter had a lower C_A and higher I values (24.96 and 125.93 %, respectively). The values of inhibitory index of untreated vine grape were very high (125.93%) due to long t_{max} and low T_{max} of hydration [1, 16].

Thus, as per the classifications of Sandermann and Kohler [21] and Hofstrand *et al*. [11] the vine grape pruning particles are extremely inhibitory. They can be classified as "unsuitable" and require special pretreatments to reduce their inhibitory characteristics with cement. Whereas the European redwood proves to be least inhibitory and may be considered as "suitable under limited conditions" for making WCP panels.

The differences in the compatibility between vine grape and European redwood under untreated conditions can be attributed to differences in the cold and hot water soluble substances of wood, determining the solubility and the pH of their extracts [14-16]. The differences in the type and quantity of hemicelluloses also matter. *Pinus sylvestris* as a softwood species should have different xylans in its polyoses, compared with the vine grape, a hardwood species. The results obtained for solubility and pH of the extracts (Table 2) are consistent with the hydration characteristics results of the untreated materials [13]. Vine grape particles contain the higher hot and cold water substances and the lower pH values than *P. sylvestris* (Table 2). Gnanaharan and Dhamodaran [13] reported that species with low acidic extract along with low cold water solubility will be suitable for wood-cement-wool board manufacture. These results are in agreement with the results of research carried out in other parts of the world [32, 33].

Effect of Treatments on the Compatibility of Vine Particles with Cement: Analysis of variance revealed that the treatments used significantly affected all hydration parameters of wood-cement mixtures. Interaction of kind X treatments was also significant. This means that each lignocellulosic material reacted differently when mixed with cement. Figure (2) shows the effect of different treatments on hydration characteristics of the vine grape and European redwood.

For each lignocellulosic m aterial there was a certain combination that produced the best result [1, 4, 10]. Use of cold or hot water extraction for the two materials used caused enhancement in their compatibility with cement depending on the types of materials, which is evident from changes in the hydration curves (Fig. 2), increase in T_{max} and C_A values and decrease in t_{max} and I values of the wood-cement mixtures (Table 3 and Fig. 2).

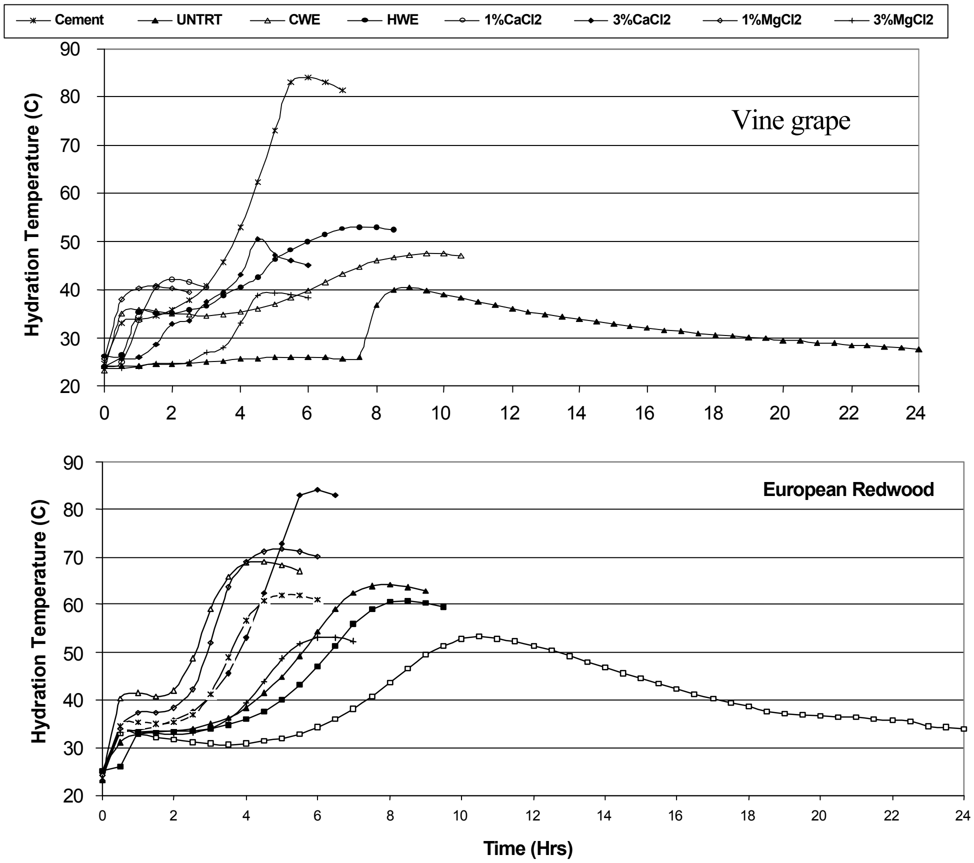

Fig. 2: Exothermic hydration curves of the mixtures of the vine and the European redwood particles with cement under different treatments, as compared with net cement. For explanations see table 3

Table 3: Maximum temperature (T_{max}), time to reach the maximum hydration temperature (t_{max}), inhibitory index (I) and compatibility factor (C_A) for the mixtures of vine particles-cement and European redwood-cement as affected by different treatments

Species	Treatments							
	Code	Wood	Additives	T_{max} (°C)	.t_{max} (.h)	I (%)	C_A (%)	Status*
Vine particles	UNTRT	None	None	40.33	24.00	125.93	24.96	Unsuitable
	CWE	CWE	None	47.50	9.39	15.99	52.34	SU
	HWE	HWE	None	49.33	7.56	8.65	56.45	SU
	1%CaCl2	Untreated	1%$CaCl_2$	43.43	2.00	95.35	26.28	Unsuitable
	3%CaCl2	Untreated	3%$CaCl_2$	50.27	4.50	6.66	69.37	SU
	1%MgCl2	Untreated	1%$MgCl_2$	40.77	1.50	108.11	37.67	Unsuitable
	3%MgCl2	Untreated	3%$MgCl_2$	39.27	5.00	104.11	29.84	Unsuitable
European	UNTRT	None	None	52.50	9.93	13.24	78.49	SU
Redwood	CWE	CWE	None	59.83	7.99	4.34	81.74	Suitable
	HWE	HWE	None	62.67	8.23	4.41	86.44	Suitable
	1%CaCl2	Untreated	1%$CaCl_2$	63.67	5.04	-1.02	82.01	Suitable
	3%CaCl2	Untreated	3%$CaCl_2$	69.73	4.58	-0.56	90.52	Suitable
	1%MgCl2	Untreated	1%$MgCl_2$	55.37	6.23	-0.77	89.96	Suitable
	3%MgCl2	Untreated	3%$MgCl_2$	69.53	4.98	-0.39	92.94	Suitable
LSD0.05 for species *Treatments				3.95	2.54	9.71	7.52	
Net cement				83.6	6.28	0.00	100.00	

Each value represents the mean of three replications

CWE: Wood extracted by cold water. HWE: Wood extracted by hot water

SU: Suitable under limited conditions. * According to Sandermann and Kohler (1964)

For neat Portland cement: T_{max} (83.6°C), t_{max} (6.28 h), I (0) and C_A (100%)

(-) Negative I values due to lower t_{max} of neat cement than t_{max} of wood-cement

There were large variations in the enhancement obtained by using the cold or hot water extraction for the vine grape and the European redwood. Generally, hot water extraction was more effective than cold water extractions [10, 19].

Compatibility of Wood-cement Mixtures Affected by Pretreatments: Under cold or hot water extraction and based on the classification of Sandermann and Kohler [21] and Okino *et al*. [17, 29], vine grape particles were suitable under limited conditions only if they were treated with hot water since T_{max} stayed between 50 and 60°C (52.33°C) and C_A value increased to 56.45%. On the other hand, European redwood was elevated from 'suitable under limited conditions' to 'suitable' for making wood-cement board by using either cold or hot water extraction (Table 3). This improvement can be attributed to removal of sugars and other water-soluble substances from woody and non-woody materials used, especially for vine grape, which appears to be highly inhibitory to setting of cement in the natural state. Addition of cold or hot water extracts of these materials to the cement endorsed these inferences. The inhibitory effect of these extracts on cement setting can be noted by the increase in t_{max} and I values and decrease in T_{max}, ΔT and C_A values [10, 15, 16].

Chemical Additives and Compatibility of Wood-Cement Mixtures: With regard to the addition of either $CaCl_2$ or $MgCl_2$ (1 or 3% based on cement weight) to the mixture of cement and untreated vine grape and European redwood, it is obvious from Table (3) and Figs. (2), that each material reacted differently with cement when added with chemical additives. The highest T_{max} values were obtained for untreated European redwood-cement mixture after adding 3% $CaCl_2$ or $MgCl_2$ (69.73 and 69.53°C, respectively). Based on the classification of Sandermann and Kohler [21] and Okino *et al*. [29], vine grape were reclassified from 'unsuitable' to 'suitable under limited conditions' only if added with $CaCl_2$, whereas European redwood was reclassified from 'suitable under limited conditions' to 'suitable' for making WCP by addition of $CaCl_2$ or $MgCl_2$ to the untreated wood-cement mixtures. No improvements could be achieved by adding 1% $CaCl_2$ or $MgCl_2$ (1 or 3% by cement weight).

Although, addition of chemical additives to wood-cement mixtures in the current study improved the compatibility with cement, these additives did not appear to have neutralized the detrimental effect of high inhibitory vine grape on exothermic reactions of cement. This is in agreement with Moslemi *et al*. [10, 33] who found that the addition of 3% $CaCl_2$ to the mixture slightly improved the maximum hydration temperature for the untreated cotton stalks and bagasse particles by 17.6% and 18.49%, respectively. However, it contradicts with Nasr [34] who reported that of the different chemical additives ($CaCl_2$, $FeCl_3$, $MgCl_2$ and NaOH), addition of 3% $CaCl_2$ to untreated cotton stalks-cement mixture caused the minimum decline in I values.

As the chemical additives act as accelerators in the case of European redwood, the improvements in various hydration parameters obtained in the current study can be attributed to speed up the rate of hydration of plain cement without reacting with the wood substances when combined with low inhibitory materials or to provide a more suitable pH for setting the wood-cement mixtures [10]. These results are in agreement with those of Zhengtian and Moslemi [32], Nasser [16], Mohamed [33] and Olorunnisola [35].

Among all treatments used in this study, a certain combination produced the best result for each material used. In the case of European redwood, all the treatments resulted in almost high improvements, but addition of 3% $CaCl_2$ or $MgCl_2$ to the untreated wood-cement mixtures gave the best results. However, out of the seven treatments used, only the addition of 3% $CaCl_2$ showed the best results for vine grape. Under this treatment, only vine grape can be used for making wood-cement particleboard. Therefore, it may be concluded that vine grape particles were suitable to produce wood-cement particleboard only if the particles were treated with hot water extraction or with addition of 3% $CaCl_2$ to the mixture. More experiments are needed to enhance the compatibility of vine grapes and cement for the wood-cement panels industry.

CONCLUSIONS

The following conclusions may be drawn from the present study:

- Vine pruning particles are extremely inhibitory which can be classified as "unsuitable" and require special treatments to reduce their inhibitory characteristics with cement. However, European redwood is least inhibitory and may be classified as "suitable under limited conditions" for making WCP.
- The best treatments, which proved to be effective for enhancing the compatibility of vine pruning particles with cement, were either extraction with hot water or addition of 3% $CaCl_2$ as accelerator. We suggest the addition of 3% $CaCl_2$ to the particles pre-treated by hot water extraction for obtaining the best result.

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