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Cement-bonded composite boards with arhar stalks

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Abstract

A study was carried out to explore the possibility of making cement-bonded composite building products using arhar stalks. The water extractive of arhar stalks is slightly acidic, pH value around 6.5. It was found that the extractive adversely affect the cement hydration and strength development processes. The increase in setting time varied from 25 to 130% when extractive content was from 0.5 to 2% by weight of cement. 28-day compressive strength reduced by 13 to 20% at 1–2% concentration of extractive powder. Further, studies showed that these effects could be overcome by adopting suitable measures like cold water extraction and/or by use of an accelerator. A dose of 2% calcium chloride, by weight of cement, could offset the effect of 1% concentration of arhar extractive. The cement bonded composites were prepared by varying the flakes content from 0 to 32%, by weight, in the flakes–cement mix using a casting pressure of 3 MPa and demoulding time up to 10 h. The results showed that cement composites with bending strength >9.0 MPa and internal bond strength >0.6 MPa could be made using arhar stalks as a reinforcing material. The strength properties of arhar stalks–cement composites were found to satisfy the minimum requirements of International Standard, ISO:8335-1987.

Keywords: Cement; Composite; Arhar; Boards; Setting; Extractives

1. Introduction

Arhar is one of the popular pulses used as a main food item in many south Asian countries like India, Pakistan, Bangladesh, Sri Lanka and others. The plant is also grown in Africa, Australia, America, East and West Indies, Malaya and Netherlands. Arhar stalks are abundantly available as an agricultural residue from the plant. Its botanical name is *Cajanus cajan* L. Millsp.

Arhar is an annual crop sown at the beginning of rainy season (June/July) in India and harvested in winter (November–December). The plant is grown in most parts of India. After taking out the arhar for making pulses, the stems and branches of its plant (arhar stalks) remain as by-product in large quantity. These stalks are up to 3 m high and 75 mm in diameter and are disposed by various means. The stalks used in the present study are shown in Fig. 1. Generally, the thinner sections of stalks are used as a fuel and the thicker sections are used for making temporary shelters in rural areas, mainly for roofs. The stalks are placed in layers over purlins and rafters for making roof covering, which is further covered with locally available grasses or encased in a mud matrix. The dry stalks are cheap, strong and durable and thus can be a prospective reinforcing material for cement based building products.

Studies have been carried out to see the effect of water soluble compounds present in wood and natural fibres on the hydration and strength development of cement composites. It has been reported that the water-soluble extractives of wood retard and sometimes inhibit the normal setting and strength development properties of cement during the production of wood–cement composites. A similar effect of delay in setting of cement has been observed when composites are made using plant fibres. Studies have shown that hemicelluloses, starch, sugar, tannins, certain phenols and even lignins are responsible for this. These extractives affect the fibre–cement composites in two ways i.e., they produce delay in setting of cement and retard the strength

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Fig. 1. Dried arhar plant.

development in the composite material [1–7]. This prolongs the demoulding time of shaped units and affects the production rate. Soaking of fibres in water and/or use of chemicals like aluminium sulphate, magnesium chloride, calcium chloride is recommended to overcome this problem.

Investigations have been carried out on the use of wood and natural fibres like coir, sisal, jute, sunhemp, bagasse etc. for the development of cement bonded composite building products. The major focus was to replace asbestos by these eco-friendly materials for producing fibre–cement composites. These studies resulted into the development of different types of building products, mostly boards and tiles, having varying density and physico-mechanical properties depending upon the nature of fibre, mix design and processing parameters. The developed composite products are being used by the building industry in place of asbestos based products.

Durability studies revealed that fibre–cement composites loose strength and ductility on out side exposure, which is probably due to the degradation of fibres in highly alkaline environment of cement matrix. Gram [8] reported that due to the high alkaline environment of the cement matrix the sisal fibre cement composites became brittle when stored outdoors. It has been postulated that this may be due to the reaction of alkaline pore water with the lignin and hemicelluloses existing in the middle lamellae of fibre cells (weakening the link between the individual fibre cells) or densification of matrix in the fibres due to the gradual filling of the fibre cell cores with the hydration products (brittle breakage of fibre under stress). Durability of fibre–cement composites can be improved by reducing the alkalinity of cement matrix. Rahim and Swamy [9,10] reported that durability of oil palm fibre cement composite products can be improved by replacing a certain proportion of ordinary Portland Cement by pulverised fly ash (PFA), rice husk ash (RHA) or latex.

The objective of this study was to find out the possibility of using arhar stalks for making cement bonded composite products.

2. Raw materials

Arhar stalks were obtained from the local farmers. These stalks were kept in open for sun-drying for about one month. The stalks cannot be used as such for making building boards and need to be converted either into particles, flakes or pulp. Hence, the stems of the arhar stalks were cut into smaller pieces of size 100–150 mm in length. These smaller cut pieces were soaked in water for 2 h at room temperature to soften the stalks and then passed through pressure rollers in a two roll mill with incremental increase in pressure between rollers. The material is converted into flakes first and then into particles.

The pulp was prepared by the soda process in which 15% (wt.) of sodium hydroxide was used to digest the material under a pressure of 80 psig (0.55 MPa) and at 125 ± 2 °C for 60 to 90 min and then cooled and washed with fresh water [11]. The pulp yield on dry weight basis was $28 \pm 2\%$.

Ordinary Portland cement was used as binding material. For making cement mortar local river sand was used, which is generally used in making cement mortar and concrete in the local construction works. Calcium chloride of analytical reagent grade was used as cement setting accelerator.

In an investigation three forms of arhar were used for making composite boards by varying the composition of the mix, casting pressure, and demoulding time [11]. The results revealed that the composite boards prepared by using arhar stalks and pulp showed better properties and meet the minimum standard requirements. Further, composite boards from arhar flakes were found cost effective.

3. Experimental methods

3.1. Properties of arhar stalk particles

Bulk density (BD) of the arhar particles (stalk, flakes or powder) was determined by filling them in a beaker of volume of 500 cm^3 with occasional tapping and weighing them. BD in g/cm^3 was calculated by dividing the weight

Table 1
Properties of different forms of arhar stalk

Property	Arhar Stalks			
	Stalk	Flakes	Powder	Pulp
Bulk density, kg/m^3	200–250	85–120	150–225	65–100
Moisture content, wt.%	10.82	10.10	10.90	6.10
Water absorption (24 h), wt.%	55–65	170–200	250–320	250–400
pH	6.4	6.6	6.3	7.1
Cold water extractives (24 h), wt.%	1.0–2.0	1.5–2.5	3.0	–

in gram of the arhar particles by the volume i.e. 500 cm^3. The weight of arhar particles was measured with moisture content at 25 ± 2 °C at 60 ± 10% humidity. The value of BD was subsequently converted from g/cm^3 to kg/m^3 by multiplying a factor of 1000. For measuring the moisture content of the arhar particles, a small sample of about 10 g was weighed (W_1) and kept in an oven at 100 ± 2 °C. The samples were taken out at regular intervals and weighed. It was repeated till a constant weight was obtained (W_2). Moisture content (m) in percentage was obtained using the following relationship:

$$m = \frac{W_1 - W_2}{W_2} \times 100 \quad (1)$$

To find out pH values and the amount of cold water soluble extractives 50 g (W_3) of oven dried arhar stalk, flakes or powder was dipped in beakers containing 750 ml of distilled water, required to completely immerse the particles [7]. The soaked particles were filtered after 24 h and washed with distilled water. 50 ml of the filtrate is taken in a beaker and its pH was determined using a pH meter. 500 ml of the filtrate taken in another beaker and dried in an oven at a temperature of 60 ± 2 °C and weighed till it reached a constant weight (W_4). The amount of cold water solubles in 24 h in percentage (C_e) was then calculated using the following relationship:

$$C_e = \frac{W_4}{W_3} \times \frac{750}{500} \times 100 \quad (2)$$

The arhar particles obtained after filtration in the above test were weighed (W_5) after draining off the excess water and the water absorption value in percentage (w) was then computed with the following equation:

$$w = \frac{W_5 - W_3}{W_3} \times 100 \quad (3)$$

Four replicates were used to determine the above properties and the mean value of the results obtained is reported in Table 1.

3.2. Extractive content

The rate of extractive leaching from the three different forms of arhar i.e. stalk, flakes and powder was determined by dipping a sample of 50 g of arhar in beakers containing 750 ml of distilled water, required to completely immerse the stalks [7]. The dipped arhar particles were removed from the beakers and washed with distilled water after 1, 3, 7, 10, 15 and 30 days. After filtering a fresh 750 ml of distilled water was filled into the beakers containing the arhar. The filtrate was dried in oven at a temperature of 60 ± 2 °C and weighed.

3.3. Cement properties

The dried extractives were mixed with cement in various proportions e.g. 0.5, 1.0, 1.5 and 2.0% by weight of cement and the mixes were used for determination of setting times and compressive strength. The extractive was added in desired quantity to half of the mixing water and thoroughly mixed. Then the solution was poured over the cement or cement-sand mixture and mixed. This was followed by addition of the remaining amount of water and subsequent mixing. Initial and final setting times of neat cement paste and extractive-cement mixes were measured by using Vicat apparatus. For determination of compressive strength cubes of 50 mm size of cement-sand mortar (1:3) were cast. The strength was measured at 1, 3, 7 and 28 days. Similar studies were carried out by mixing accelerator (0.5 to 2.0% by weight of cement) along with the extractive.

There were two main reasons that led to adopt this approach of using extractives instead of arhar particles. The first is non-availability of a standard procedure to determine setting times of cement containing fibres or organic particles and the second is that the presence of arhar particles can affect compressive strength of cement. Thus, it is difficult to ascertain whether the effects are due to extractives or due to the presence of arhar particles or combined effect on strength. Previously also various researchers adopted this kind of approach. For example, Zhengtian and Moslemi [3] extracted the water-soluble compounds using a soxhlet extractor from western larch and studied the effect of extraction period on the hydration of wood–cement mixtures, whilst Miller and Moslemi [12] used model compounds that are generally found in wood to study the effects on hydration and tensile strength of cement.

3.4. Composite boards

3.4.1. Preparation

Composite board specimens were prepared by varying the flakes content (0 to 32% by weight) at a casting pressure of 3 MPa and demoulding time 10 h following the procedure described here after. A weighed amount of arhar flakes, 15–20 mm length and 0.3–0.5 mm thickness, were soaked in water for 24 h after which unabsorbed water was drained off. The arhar flakes were then thoroughly mixed with predetermined quantities of cement and water. A water/cement (w/c) ratio of 0.40 was used. The extra water, in addition to the water contained in the pre-soaked particles, required to maintain the w/c ratio of 0.40 was

Fig. 2. 400 ton capacity hydraulic moulding press.

determined using the water absorption (w) of the arhar stalk particles. For example, if the mix consists of 100 g of stalks and 1000 g of cement. As per the w/c ratio of 0.40, the total water requirement is 400 g. Out of this, 185 g of water is already present in the stalk particles kept immersed in water for 24 h (as w of the stalks used in the present investigations is 185%). Therefore, an additional amount of 215 g of water is to be added in the mix.

The mix containing arhar flakes + cement + water was uniformly spread in a steel mould (300×300 mm size). The mix was pressed hydraulically using 3 MPa pressure with the help of steel plungers, so that 10 ± 1 mm thick specimen was obtained. The whole assembly of the mould and plunger was kept in the pressed position for the specified time of 10 h. A 400 tonne capacity hydraulic press used for making the boards is shown in Fig. 2. The press has 100×100 cm platform and thus panels up to 100×100 cm size can be made by using the above press at 350 T pressure. In the present investigations two boards of 30×30 cm size were pressed at a time. The pressed boards were kept in the press for 10 h, i.e, the load was kept on the boards for 10 h. There was no leaching of water or cement–water slurry from the mix when pressed at 3 MPa pressure at w/c ratio of 0.40.

This duration was optimised for the arhar flakes–cement mix to attain a strength that would not allow the mix to spring back after removal of the pressure. It means that there was no variation in the thickness of the consolidated composite mass after the pressure release, as there was no appreciable change in the thickness of the board when measured at different points after demoulding. The samples thus obtained were demoulded, moist cured for 10 days and finally subjected to air-drying for 30 days.

3.4.2. Testing

The density, water absorption, thickness swelling, bending strength and internal bond strength (tensile strength perpendicular to the surface of a board) properties of the composite board were determined to study the effects of various parameters mentioned earlier on the arhar–cement composite samples. The 10 ± 1 mm thick samples were tested for various properties following the procedures described in ISO: 8335-1987 [13] and IS: 14276-1995 [14]. Density, water absorption and thickness swelling were measured by using 100×100 mm strips, whilst 300×75 mm rectangular strips were used for bending strength test and 50×50 mm strips to determine internal bond strength of samples.

The internal bond (IB) strength was tested as tensile strength perpendicular to the surface of the composite board according to the method given in IS:14276-1995. For this test 50×50 mm size specimen were used. A steel loading block of 50×50 mm and 25 mm thick was bonded with an epoxy resin on each 50 mm square face of the specimen. After seven days of curing, the steel blocks were attached to the pulling heads of a Universal Testing Machine (Shimadzu, Model AGS2000G) and test conducted at a crosshead speed of 1 mm/min. The maximum load was divided by the area of the specimen i.e. 2500 mm^2 to obtain the internal bond strength.

The bending strength was measured in three point loading using a span of 200 mm at a mid-span deflection rate of 5 mm/min on the test machine. Modulus of elasticity was calculated from the load–deflection curve in bending. The water absorption and thickness swelling values of the composite were obtained by immersing the specimens in water for 24 h at 25 ± 2 °C and then taking measurements. At least six samples were tested for each property and the mean value is reported.

4. Results and discussion

4.1. Extractive content

The extractive leach out rate in cold water from different forms of arhar is shown in Fig. 3. It can be noticed that most of the extractives from all the three forms of arhar come out within a period of 3 h after dipping in the water (Fig. 3a). However, the leaching rate decreases with time (Fig. 3b). The process of cold-water extraction can remove inorganic compounds, sugar, gums and phenolic compounds like tannin [15]. The arhar extractives are slightly acidic as the pH value varies around 6.5 (Table 1). The colour of the extractives was reddish brown.

These leaching characteristics of arhar are similar to other lignocellulosic materials like wood, coir and sisal fibres. However, the amount of extractive appears to be relatively low in arhar stalks [7,16,17]. As can be seen in Fig. 4 that the extractive removed in one day from arhar powder is slightly less than that in coir, but considerably lower than that in sisal. This is beneficial from the arhar–cement compatibility point of view.

4.2. Setting times

The effect of extractive content on setting times of cement is shown in Fig. 5. It is evident from the figure that

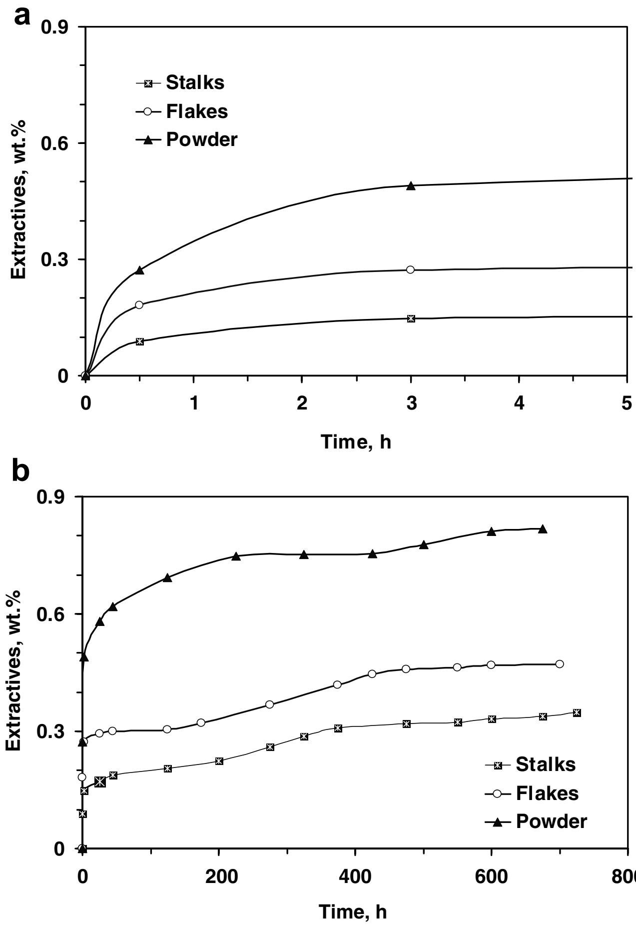

Fig. 3. Amount (by wt.% of arhar waste) of cold-water extractives removed from different forms of arhar waste. (a) Up to 5 h extraction period. (b) Up to 720 h extraction period.

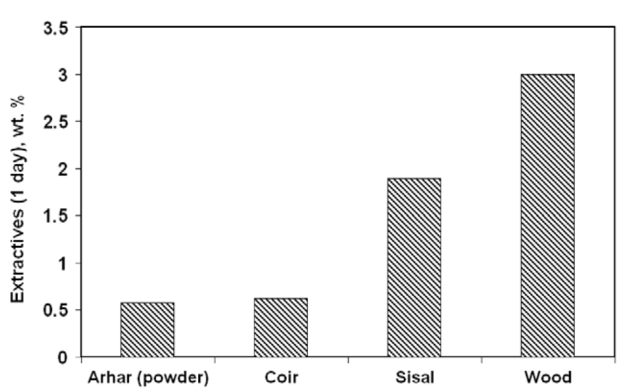

Fig. 4. Comparison of cold water extractives removed in one day in plant materials. (Data for coir and sisal taken from reference [7] and wood from reference [17].)

both initial and final setting times of cement increase with the increasing dose of extractives. The effect on initial setting time is particularly important, which increases about 25% when extractive content is 0.5%, about 58% when

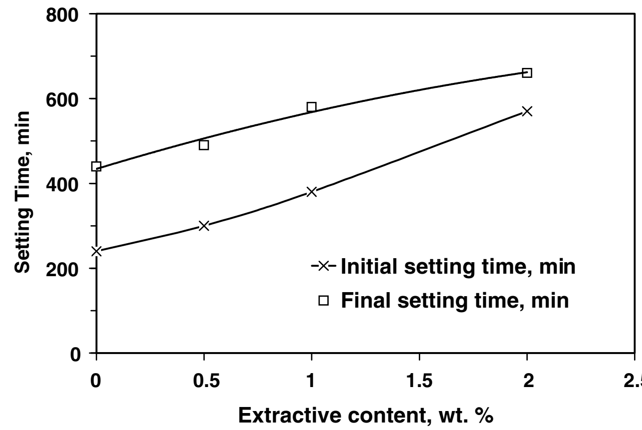

Fig. 5. Effect of arhar extractives on setting time of cement.

extractive content is 1% and more than 130% when extractive content is 2% by weight of cement. The delay in setting is due to interference of the extractives in cement hydration process. There are two mechanisms suggested for delay in setting of cement due to presence of extractives. First is that the extractives composed of various organic compounds react with the metal ions present in the cement solution. This decreases the concentration of Ca^{2+} ions in the solution and possibly disturbs the equilibrium of the solution, which delays the start of nucleation of $Ca(OH)_2$ and C–S–H gel [18–20]. Another suggested mechanism is that the organic compounds coat the hydrating cement particles and slow the hydration process [21]. Nevertheless, the delay in cement setting can be minimised by using a suitable accelerator like calcium chloride [7,20].

4.3. Compressive strength

The effect of extractive content on compressive strength of cement mortar at various periods is shown Fig. 6. The results show that the effect of extractives is more in the initial stages, but at 28 days the strength is almost similar for low concentration of extractive i.e. 0.5% by weight of cement. However, at higher concentration (1 and 2%) the effect is significant on the hydration of cement, which prevents strength gain. At these concentrations the 3-day strength reduces by 30 to 40% and 28-day strength reduces by 13 to 20%. Nevertheless, the addition of calcium chloride reduces the effect of extractive on compressive strength of cement mortar as can be seen in the Fig. 7. It can be noted that a dose of 2% of calcium chloride by weight of cement can offset the effect of 1% concentration of arhar extractives.

4.4. Properties of composite board

The effect of change in flakes content on the density, water absorption, bending strength and internal bond strength of flakes–cement samples is shown in Figs. 8–11, respectively. It can be noticed that the density of the spec-

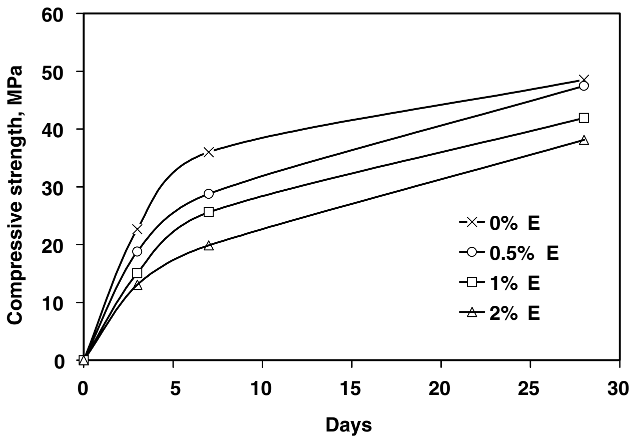

Fig. 6. Influence of arhar extractive content on strength development in a cement mortar (E = Extractive).

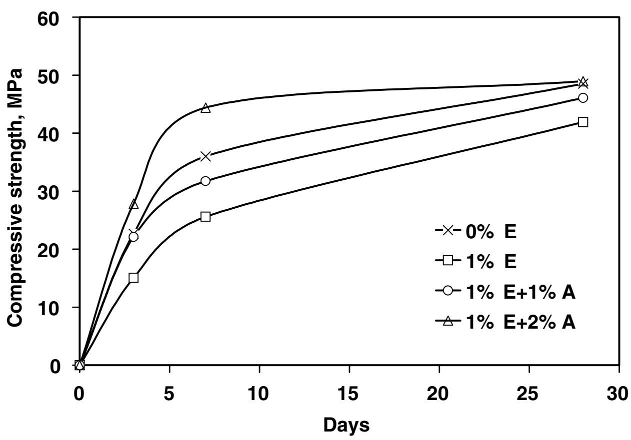

Fig. 7. Effect of accelerator (A) dose on compressive strength of cement mortar containing 1% arhar extractive (E) by weight of cement.

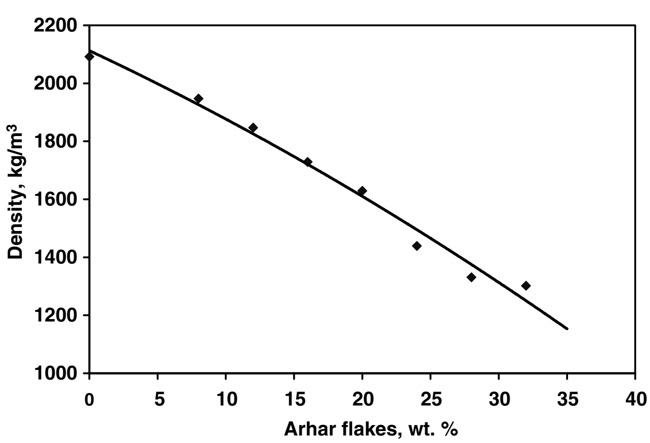

Fig. 8. Effect of arhar flakes content (% by weight of cement) on density of the composite board.

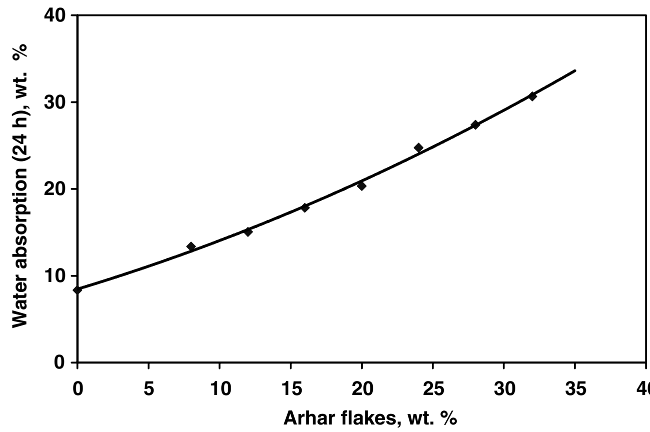

Fig. 9. Water absorption (24 h) vs arhar flakes content (%) by weight of cement.

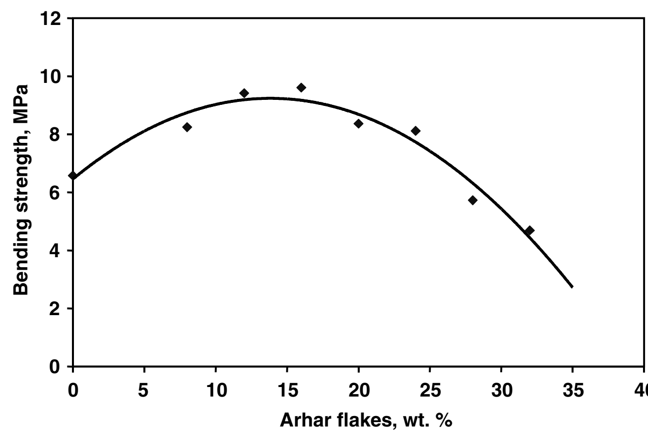

Fig. 10. Bending strength of composite board vs arhar flakes content (% by weight of cement).

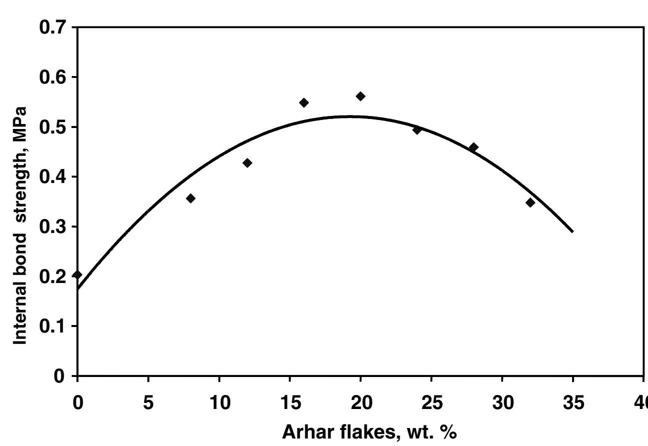

Fig. 11. Effect of arhar flakes (% by weight of cement) on internal bond strength of the board.

imens decreases with the increase in flakes content in the flakes–cement mixture (Fig. 8). The reduction in density means the board becomes more porous, which is reflected in increased water absorption (Fig. 9) trends. An increase in flakes content from 8 to 16% resulted in a decrease in density from 1950 to 1729 kg/m^3 while the water absorp-

Table 2
Comparison of composite board properties with the requirement of standards

Property	Experimental values		ISO:8335-1987 and BS: 5669-1989: Part 4 Specifications
	Arhar flakes in the mix % (wt.)		
	12	16	
Density, kg/m^3	1847	1729	1000 (min.)
Thickness swelling, %	0.09	0.10	2.0 (max.)
Bending strength, MPa	9.42	9.61	9.0 (min.)
Modulus of elasticity, GPa	4.12	3.27	3.0 (min.)
Internal bond strength, MPa	0.63	0.65	0.45 (min.)
Moisture content, %	9.05	9.84	6–12
Water absorption, %	15.05	17.83	–

tion increased from 13.4 to 17.8%. This may be due to the replacement of dense material (cement) by a porous material having higher water absorption values (flakes) in the flakes–cement mix. It can be further observed that the replacement of cement by a lower density material flakes increases the total volume of the mixture, even after compaction at 3.0 MPa pressure, and that the increase in volume is related to the amount of flakes present in the mixture. For example, when the flakes content in the mixture was increased from 8 to 16%, the total volume of the mixture showed an increase of about 12% after compaction. It is therefore concluded that, because of the increase in the volume of the mixture, the density of samples decreased, resulting in an increase in water absorption. The thickness swelling of the composite (24 h soaking) is generally less than 2% as stipulated by the standard, Table 2. It shows that the composites have good dimensional stability.

The bending strength of the flake-cement specimens increases initially with an increase in the flake content and the maximum values are obtained at flake loading between 12 and 16% by weight (Fig. 10). However, a further increase in flakes content shows a reduction in the bending strength values and at 32% flake loading, the strength value is very low i.e. 4.7 MPa. The results show that the loading of 12 and 16% flakes in a cement matrix increases the bending strength of the material up to 9.4 MPa and 9.6 MPa, respectively in comparison to 6.6 MPa at no flake loading. This increase in the strength of the composite material with an increase in the flake content is in accordance with rule of mixtures [22]. However, on further addition of flakes, i.e. up to a flake content of 24%, the bending strength reduces to 8.1 MPa. The decrease in strength at higher flake content may be due to the inefficient utilisation of flakes attributed to formation of 'clumps' and damage to flakes, which increases the porosity of the composite. It also results in the reduction in flakes-matrix interfacial area and hence lower strength properties than the expected.

In the internal bond strength most of the failures of composite board was in between the thickness of the board up to 24% of loading and the bond between the composite sample and steel block was intact. But at higher percentage of the arhar particles the failure was also observed from near the joint of the board with steel block. There was no definite trend of the failure, as sometimes the failure was from the middle of the board and sometimes from near the upper or lower portion of the board thickness. It was observed that the failure of the boards was both due to failure of fibre-to-fibre bond and fibre-to-matrix bond. At lower percentage it was predominantly fibre-to-fibre bond failure and at higher percentage it was due to fibre-to-matrix bond failure, i.e., fibre pull out from the matrix. The internal bond strength of the composite was found to be maximum when flake content was 16–20% (Fig. 11). At this concentration, probably maximum reinforcing effect of arhar can be achieved with optimum volume of cementitious matrix. Further, addition of flakes increases volume of flakes and reduces volume of matrix causing lower bond strength. Also, as the flakes content in the mix is increased, a greater number of flake-to-flake bonds are formed. The increase in flake-to-flake bonds reduce the interfacial area of contact between the flakes and the cement matrix and hence diminishes the potential of a given flake to be able to bond with the matrix. The reduction in flakes–matrix bonding would ultimately lower the strength properties. The test results of arhar flakes–cement composites showed similar behaviour, Fig. 11. From these results it appears that composites having 12–16% flakes have the optimum properties. The properties of the composite boards containing 12–16% flakes by weight of cement are compared with those specified by standards [13,23] for such boards in Table 2. It can be noted that these composite boards comply to the minimum requirements of these standards. Thus, arhar flakes can be used for making cement bonded composites products. Durability study to determine their long term performance is in progress.

5. Conclusion

The results of this study showed that the cold water extractive content in arhar stalk powder is comparable with that in coir but considerably lower than other plant materials like sisal and wood. It has been found that most of the extractives come out within three hours after immersion in water. The extractives present in arhar stalks adversely affect cement hydration and strength development processes. This problem was overcome by immersing the stalks in water for 24 h so that most of the extractives can be removed. A dose of 2% calcium chloride by weight of cement has also been found suitable to counteract the adverse effects of 1% extractives.

Cement bonded composite building boards can be produced using arhar flakes. The developed boards meet the requirements of standards on cement-bonded boards. The boards are eco-friendly as they are produced from the renewable agro-waste biomass. Therefore, in countries where arhar stalks are available, these can be used as alternate to asbestos for producing fibre–cement composite products.

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