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Microstructure and compressive strength of gypsum-bonded composites with papers, paperboards and Tetra Pak recycled materials

Dafni Foti[1], Stergios Adamopoulos[2*], Eleni Voulgaridou[1], Elias Voulgaridis[1], Costas Passialis[1], Stephen Osakue Amiandamhen[2] and Geoffrey Daniel[3]

Abstract

The incorporation of recycled papers, paperboards and Tetra Pak as filling materials in brittle matrices presents an interesting approach in the utilization of waste materials for building construction. This paper examines the compressive strength and microstructure of gypsum-bonded wastepaper-based composites. Recycled wastepaper of various types (office paper, magazine paper and newspaper), cardboards, paper boxes and Tetra Pak were shredded to short length strips of about 4 × 18 mm. The shredded materials were used as filling materials in natural gypsum in a ratio of 1:3 (v/v), and water was added to the mix. The paste was formed in cylindrical samples measuring 10 cm in length and 5 cm in diameter. Seven different types of composites were produced depending on the material used. The composite products with newspaper and magazine paper had significantly lower density and compressive strength ($p < 0.05$) than the others. However, the differences were small to have any practical importance. The density values ranged between 1.26 and 1.34 g/cm^3, and compressive strength was the lowest (4.48 N/mm^2) in the gypsum–magazine paper composites and the highest (6.46 N/mm^2) in the gypsum–Tetra Pak I composites. Since the samples produced in this study exhibited adequate compressive strength, the products could be suitable for such applications as interior walls in building constructions. Scanning electron microscopy (SEM) examination of the fractured surfaces revealed needle-like structures of gypsite crystals surrounding the fibers, which indicates good adhesion between the hydrophobic matrix and lignocellulosic fibers.

Keywords: Compressive strength, Density, Natural gypsum, Recycled wastepaper, Tetra Pak, SEM

Introduction

Lignocellulosic fibers in various products such as office paper, magazine, newspaper, cardboards, paper boxes and Tetra Pak represent a sustainable source of raw material for composite production. Tetra Pak packaging material used for food storage and preservation is composed of about 75% kraft pulp fibers, 20% low-density polyethylene (LDPE) and 5% aluminum foil by mass. Because of the extensive use of these materials, huge quantities are disposed over the world as wastes. In 2017, the recovered paper collection rate was 72.3% in the EU-28 countries plus Norway and Switzerland. The utilization rate (e.g., use of paper for recycling in the paper and board sector) in 2017 was 52.4%. This means that the total use of paper for recycling was 42.3 million tons while the total paper and board production amounted to 92.2 million tons [1]. Thus, there is likelihood for a continuous supply of raw materials for other applications than paper production. The requirement for better management and utilization of waste materials is to reduce their volume, to use and to recycle them by employing appropriate technologies of producing new products. Such technologies have been employed in inorganic-bonded wood and fiber composites [2, 3], which have been developed for several decades and are still the subject for much research with target interest in waste fiber utilization. Cellulosic fibers play a significant role in determining the properties

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*Correspondence: stergios.adamopoulos@lnu.se
[2] Department of Forestry and Wood Technology, Linnaeus University, Växjö, Sweden
Full list of author information is available at the end of the article

of fiber-reinforced inorganic composite products [4]. It has been reported that the surface characteristics and chemical composition of fibers have pronounced effects on composite performance [5, 6]. Strong interfacial adhesion between matrices and hydrophilic fibers results in improved physical and mechanical properties.

Fiber residues provided from recycled paper processing were used as additives in proportion up to 30% wt. in clay to produce fired bricks (1100 °C) that exhibited increased porosity, reduced thermal conductivity and acceptable compressive strength [7]. Demir et al. [8] used short fiber residues from kraft pulp production process in proportions of 2.5%, 5% and 10% wt. to clay and produced fired bricks (900 °C) that were effective for pore forming in clay and with acceptable mechanical properties. Fibers derived from recycling of magazine wastepaper were used to reinforce thin-sheet cement products and compared with virgin fiber composites. It was found that the properties of the composite products differ between recycled and virgin fibers, i.e., 1.5% less flexural stiffness, 15% lower toughness, 15% higher initial flexural stiffness, 35% less moisture movement, 20% lower water absorption and moisture content and 8% higher density in "cement-recycled fibers" due to fine content of these fibers [9, 10]. Kraft pulp fibers from pine, eucalyptus, wastes of sisal field by-product and banana pseudo-stem fibers were used as reinforcement materials for cement-based composites. Flexural strength and fracture toughness of all combinations were found to be sufficient for use in low-cost housing construction [11]. In "cement-recycled fibers from waste packaging boxes" panels, a decrease in density, compressive and flexural strength by increasing the fiber content from 0 to 16% was observed while an improvement of thermal insulation properties was found [12].

Gypsum has been extensively used in building applications and is completely recyclable. Neat gypsum is brittle and most of the times sandwiched between thin sheets of paper or blended with paper pulp. Highland American Corporations in N. America make the commercial building product, Gypsonite®, from natural gypsum and pulp from recycled newspapers. Experimental gypsum–wood fiber composite panels, 8 mm thick, were produced by using different types of gypsum (natural and recycled) and kraft pulp fibers from recycled cement bags with an addition of 10% by mass of limestone [13]. The addition of fibers in proportion of 12.5% by mass gave the highest values of modulus of rupture which was 160% higher compared to samples without fibers. Cylindrical samples were produced from gypsum, water and fibrous materials from reclaimed newspaper and paper pulp in proportions of 10–30% for newspaper and 5–30% for paper pulp (w/w), and tested for determination of their compressive strength [14]. The density of samples for both gypsum–paper pulp and gypsum–newspaper combinations decreased by increasing the content of fibrous material. Compressive strength for gypsum–paper pulp samples ranged between 0.28 and 1.65 N/mm^2 and had positive relationship with density. The compressive strength values for gypsum–newspaper composites were 2.18 N/mm^2 for 10% fiber proportion. For 20% and 30% fiber proportions, a continuous deformation of the samples was observed without collapse to occur up to the theoretical point of disappearing the sample voids [14]. Foti and Gallis [15] used gypsum, water and 20% (w/w) fibrous materials from reclaimed newspaper to produce non-fired solid bricks measuring 18.5 × 8.5 × 5.5 cm^3 in dimensions. Compression loads applied on lateral surfaces of bricks of 0.65 g/cm^3 density caused continuous deformation as described above. The values obtained from the density and compressive strength of the studied samples showed that the brick products could be used for construction of inner walls in natural buildings [14, 15].

Research on utilization of recycled Tetra Pak wastes with inorganic materials to produce composites is limited. Tetra Pak particles or fibers have been combined with cement concrete and polymer concrete to investigate the mechanical and other properties of the composite products. Martinez-Barrera et al. [16] added Tetra Pak fibers in proportions of 3.5–7% by mass in cement concrete and found a decrease in strength with increasing proportion of fibers. Composite products made from Tetra Pak particles of three sizes (0.85–2.36 mm) that were added in polyester concrete in various proportions up to 6% by mass showed that flexural and compression strength decreased with increasing fiber content and that larger particles increased the strength [17]. Also, in composites made by incorporation of Tetra Pak fibers (1–6% by mass) in polyester concrete, the compression and flexural strength were found to decrease with increasing fiber content [18].

Scanning electron microscopy has been used to study fiber-based composites. Some of these studies have been conducted to investigate the behavior of fibers, the fiber fracture mechanisms and the microstructure of the cement [4, 19, 20] or gypsum [21–24] used as matrices. These studies characterized the microstructural properties of the fiber–matrix interactions. In view of the foregoing studies, the aim of this research was to comparatively investigate the compressive strength of composite products manufactured by incorporating different fiber-based materials into natural gypsum, for use as interior walls in building applications. The microstructure of the fractured surfaces of the composites was also examined.

Materials and methods

Materials

The materials used for the experimental composites were the natural gypsum as matrix and various recycled fibers, which were used as filling materials. The filling materials were as follows:

i. Papers, which includes: (a) office paper collected from old files, photocopies, student's examination tests, corresponding letters, envelopes, etc., (b) newspaper collected from old newspapers, (c) magazine paper collected from old magazines and periodicals, (d) testliner, kraftliner and fluting papers used in corrugated board manufacturing which are available from paper and corrugated board industries as scrap materials.
ii. Paperboard and corrugated board, which originated from packaging materials. These types of boards are available in huge quantities as scrap materials in corrugated board industries and from recycling processes of municipal wastes. All the above papers and paperboards used in this study were of various grades and grammages and free of contaminants.
iii. Tetra Pak packaging materials classified as Tetra Pak I, which was collected from milk packaging and Tetra Pak II collected from packaging material of fruit and tomato juices. The difference between the two categories is the existence of an interior aluminum layer in Tetra Pak II. Tetra Pak materials can be collected as scrap materials from production and processing industries as well as waste materials from recycling bins. These materials were gently washed to remove food residues.

Experimental

The filling materials were reduced to short length strips of size 4 × 18 mm in a crosscut shredder device in order to be suitable for use in preparation of the experimental composites (Fig. 1). Bulk densities of the filling materials were determined. The shredded material and natural gypsum were mixed in a ratio of 1:3 (v/v), and the necessary amount of water was gradually added to the mix and stirred until a homogenous paste was formed. That means that the necessary amount of water was not precalculated, but it was gradually added during the experiment. The paste was poured into five cylindrical molds, representing five replications. The dimensions of the mold was 10 cm in length and 5 cm in diameter, according to ASTM C31/C31M [25] standard practice for making and curing concrete test specimens in the field. The formed samples were left to dry to constant weight in ambient conditions. Thereafter, the molds were removed, the air-dried samples weighed and the dimensions of cylindrical samples measured for determination of their densities.

Compression test of the cylindrical samples was carried out with an Amsler universal testing machine according to ASTM C39/C39M [26]. The samples were loaded in axial compression at a crosshead speed of 0.5 mm/min with maximum stress at failure and compressive strength determined. The microstructure of the fractured surfaces of the samples was observed using a XL30 ESEM (Thermo-Fisher) with secondary electrons in conventional mode at 10–20 kV. Representative specimens were carefully taken from the fractured surfaces of the samples with a forceps and were mounted on pin stubs and sprayed with gold using a high vacuum Emitech K550X sputter coater prior to imaging.

The data were analyzed using a single factor ANOVA with five replications. Mean values were separated where significant differences occurred using a two-sample *t* test (t_{cal}) assuming equal variances.

Results and discussion

Density and compressive strength

The results of the experimental gypsum-filling material composites are presented in Table 1. The table presents the data for the densities and compressive strengths of the composite products, as well as the bulk densities of the different filling materials and the amount of water required for mixing the materials. Statistical analysis showed that the composite products with newspaper and magazine paper had significantly lower density ($p < 0.05$, Table 1). However, the differences were small and probably with little practical importance, and the density of the various types of composites tested ranged between 1.26 and 1.34 g/cm^3. This presumably reflects the fixed ratio of gypsum-recycled material by volume, the narrow range of the bulk densities of the filling materials and the small differences for water required for mixing. The ratio of gypsum/recycled material was 3:1 by volume, the bulk densities of the filling materials ranged between 0.094 and 0.115 g/cm^3, and the amount of water added was practically the same (gypsum/water ratio 1.96–2.12). The bulk density of the gypsum used was 0.969 g/cm^3.

Compressive strength values did not differ considerably among the composites and ranged between 4.48 and 6.46 N/mm^2 for the different products tested. The composites with lower density using newspaper and magazine paper as filling materials had significantly lower compressive strength ($p < 0.05$). Gypsum–magazine paper composites had the lowest mean value of about 4.48 N/mm^2, while gypsum–Tetra Pak I composites had

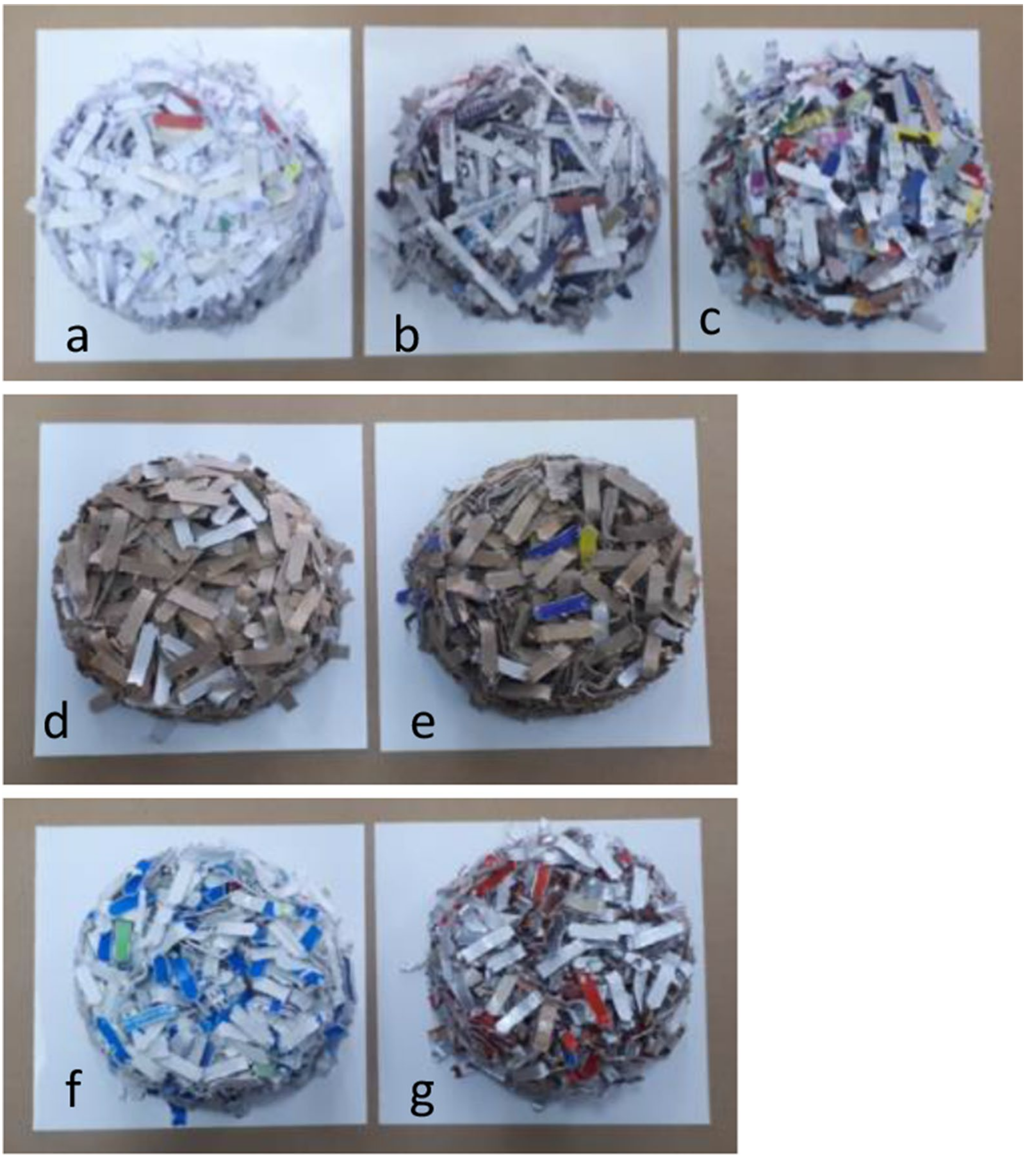

Fig. 1 Filling materials: **a** office paper, **b** newspaper, **c** magazine paper, **d** testliner, kraftliner and fluting papers, **e** paperboard, **f** Tetra Pak I, **g** Tetra Pak II

the highest mean value of 6.46 N/mm^2. In the case of "gypsum–magazine paper" where the lowest compressive strength was observed, the gypsum/water ratio needed was the lowest (1.96) due probably to low water absorptivity of the filling material as a result of heavy surface coating applied for the production of this type of paper. The low water absorptivity may adversely affect the adhesion between gypsum and magazine paper resulting in low compression strength.

Previous work on "gypsum 80%–newspaper 20%" (w/w) cylindrical samples and building bricks of low density (0.63–0.65 g/cm^3) showed that the bricks had satisfactory compressive strength (2.81 and 3.28 N/mm^2, respectively) when used as interior walls in natural building construction [14, 15]. The experimental "gypsum–paper" products in this work with higher density (1.26–1.34 g/cm^3) also exhibited adequate compressive strength (4.48–6.46 N/mm^2) for use in a similar application. Specifically, it is possible to manufacture interior walls with dimensions $300 \times 100 \times 10$ cm^3 (height, length, width) with every examined "gypsum–paper" product as revealed by the comparison of their compressive strength with the respective maximum

Table 1 Density, compressive strength, bulk density and gypsum/water ratio of different "gypsum–paper" cylindrical samples

Material	Density (g/cm^3)	Compressive strength (N/mm^2)	Bulk density (g/cm^3)	Gypsum/water ratio (w/w)
Office paper	1.30 ± 0.02^a	5.84 ± 0.25^a	0.100	2.12
Newspaper	1.26 ± 0.02^b	4.78 ± 0.24^b	0.094	2.12
Magazine paper	1.28 ± 0.02^b	4.48 ± 0.25^b	0.116	1.96
Corrugated board paper	1.30 ± 0.01^a	5.93 ± 0.37^a	0.095	2.12
Corrugated paperboard	1.30 ± 0.01^a	6.35 ± 0.32^a	0.098	2.12
Tetra Pak I	1.29 ± 0.03^a	6.46 ± 0.43^a	0.108	2.12
Tetra Pak II	1.34 ± 0.01^c	6.18 ± 0.64^a	0.115	2.12
F-value	8.148*	20.889*		

Mean values and standard deviations (±) of five replicates

* Mean values in the same column followed by different superscript letters ("a," "b," "c") are significantly different (ANOVA and two-sample *t* test, $p < 0.05$)

Table 2 Compressive strength of cylindrical samples compared with the maximum load applied on the base of an interior wall made of the corresponding experimental material

No.	Cylindrical samples			Interior wall 300 × 100 × 10 cm^3 (height, length, width) = 300,000 cm^3 volume	
	Material	Mean density, (g/cm^3)	Compressive strength, (N/mm^2)	Weight of wall, (kg)	Load applied by the wall itself on the base of wall, (N/mm^2)
1	Gypsum–office paper	1.30	5.84	390	0.038
2	Gypsum–newspaper	1.26	4.78	378	0.037
3	Gypsum–magazine paper	1.28	4.48	384	0.038
4	Gypsum–corrugated board paper	1.30	5.93	390	0.038
5	Gypsum–corrugated paperboard	1.30	6.35	390	0.038
6	Gypsum–Tetra Pak I	1.29	6.46	387	0.038
7	Gypsum–Tetra Pak II	1.34	6.18	402	0.039

load applied on the base of the wall (Table 2). Due to small differences in compressive strength of the gypsum composites and bulk densities of the filling materials used, it is suggested for production purposes that all different types of filling materials can be utilized as a mixture. However, Tetra Pak materials need to be recycled separately since they had to be washed to remove the traces of food contaminants. It should be noted that the shredded materials were added to gypsum as filling and not as reinforcing materials. This work was focused on whether the tested products "gypsum-filling materials" exhibit adequate mechanical

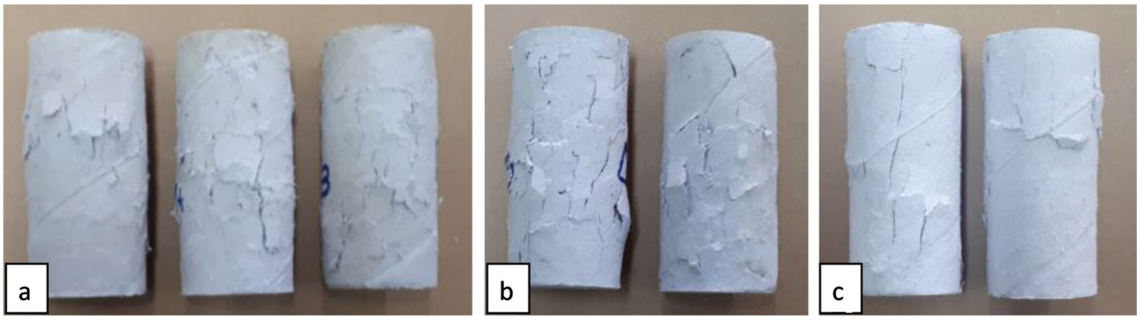

Fig. 2 Fracture types of the seven "gypsum-filling materials" combinations tested. From left to right: **a** office paper, newspaper, magazine paper, **b** testliner–kraftliner–fluting papers, paperboard, **c** Tetra Pak I, Tetra Pak II

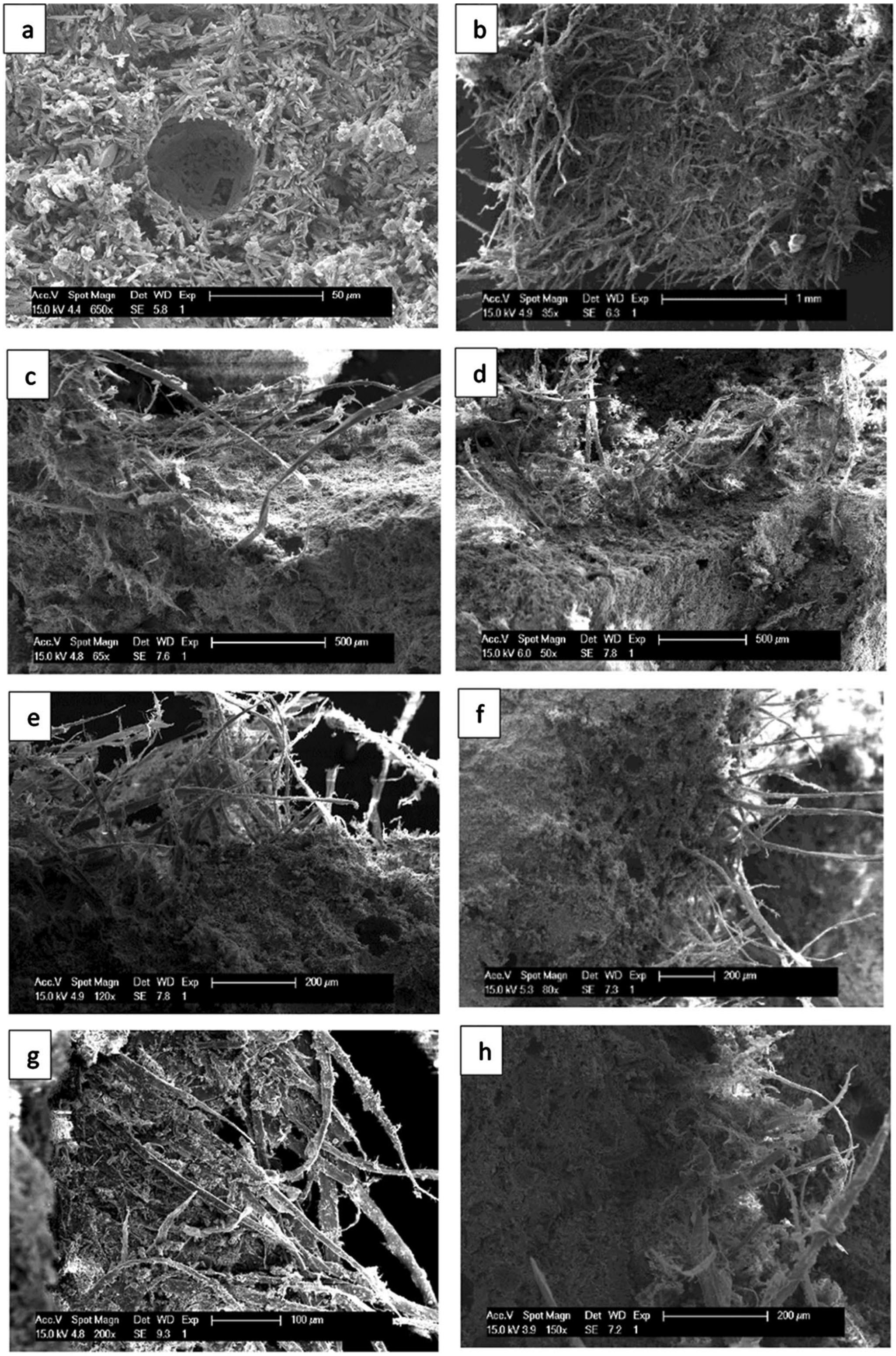

Fig. 3 SEM images showing **a** air pocket in the gypsum matrix and needle-like structure of gypsite crystals and matrix–fiber interface in (**b**) office paper–gypsum sample, **c** newspaper–gypsum sample, **b** magazine paper–gypsum sample, **e** corrugated board paper–gypsum sample, **f** corrugated paperboard–gypsum sample, **g** Tetra Pak I–gypsum sample, **h** Tetra Pak II–gypsum sample

properties to be used as building materials. It was not an objective to find how the different papers contribute to compressive strength. Fracture types of the various combinations are shown in Fig. 2. A similarity of failure was observed in all cylindrical samples, which failed in shear with planes parallel to sample axis.

Microstructure

Figure 3a shows the needle-like interconnecting structures of gypsite crystals, which are largely dihydrate crystals [13]. The morphology of the gypsite crystals is due to paste hydration when gypsum reacts with water. At the fractured surface in each gypsum–fiber sample (Fig. 3b–h), the presence of fiber pullout indicates a good level of matrix–fiber adhesion as observed by Savastano et al. [19, 20]. Similarly, the presence of crystal incrustations on the different paper fiber surfaces (Fig. 3b–h) indicates that there is good interfacial interaction between the matrix and the fibers, which improves composite's performance. The study revealed that the recycled wastepapers present good adhesion to the gypsum matrix, although with possible exception for magazine paper type (Fig. 3d).

For a brittle matrix like gypsum, it is expected that fibers may pull out under failure load. The important interfacial properties required for developing durable and tough composites are the debond fracture surface energy and the interfacial shear sliding stress (τ). The debond fracture surface energy must be considerably lower than the fiber fracture surface energy for the composites to be non-brittle. If τ is too high, the matrix micro-cracking level may approach ultimate tensile strength. This shortens the fiber pullout lengths and the composite becomes brittle. If τ is too low, the transfer of load from matrix to fiber will also be low. This results in a low micro-cracking stress, low ultimate strength and less effort on fiber pullout [27–29].

Conclusions

From the results of this study, the following conclusions can be drawn:

Small differences were found in density of the various types of composites tested, especially for these containing newspaper and magazine paper. Density values ranged between 1.26 and 1.34 g/cm^3. This could be due to the fixed ratio of "gypsum–paper material," small differences of bulk densities (0.094–0.115 g/cm^3) and the almost equal quantity of water added (gypsum/water ratio: 1.96–2.12). The mean compressive strength values did not differ considerably between the seven types of composites tested and ranged between 4.48 and 6.46 N/mm^2. The relatively low-density composites with newspaper (1.26 g/cm^3) and magazine paper (1.28 g/cm^3) had a significant lower compressive strength. The lowest value (4.48 N/mm^2) refers to "gypsum–magazine paper" and the highest value (6.46 N/mm^2) was obtained in "gypsum–Tetra Pak I" composites.

All the experimental composites exhibited adequate compressive strength for use as building bricks of interior walls in natural building constructions. The compression fracture type was observed to be in shear and was similar for all combinations tested. Due to small differences in compressive strength of the gypsum composites and bulk densities of the filling materials studied, the different filling materials can be utilized interchangeably as raw materials to produce gypsum-bonded composite products. It is recommended that Tetra Pak wastes need to be recycled and re-used separately.

SEM study revealed the presence of crystal incrustations on fiber surfaces. This indicates that there is good interfacial adhesion between the matrix and the fibers, which subsequently improves the composite's performance. Sample failure was observed to occur predominantly by fiber pullout.

Abbreviations

ANOVA: Analysis of Variance; ASTM: American Society for Testing and Materials; ESEM: Environmental scanning electron microscopy; LDPE: low-density polyethylene; SEM: Scanning electron microscopy.

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Authors' contributions

SA conceptualized the project and supervised the microscopic study. DF prepared and tested the experimental samples. EV, EV and CP supervised the composite preparation and testing. SOA and GD performed the SEM analysis. DF and SOA wrote the manuscript. SA, EV, EV, CP and GD reviewed and edited the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials

All data generated and analyzed during this study are included in this published article.

Competing interests

The authors declare that they have no competing interests.

Author details

[1] Department of Forestry and Natural Environment, Laboratory of Forest Utilization, Aristotle University of Thessaloniki, Thessaloniki, Greece. [2] Department of Forestry and Wood Technology, Linnaeus University, Växjö, Sweden. [3] Department of Forest Biomaterials and Technology, Swedish University of Agricultural Sciences, Uppsala, Sweden.

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