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The Effects of $CaCO_3$ Coated Wood Free Paper Usage as Filler on Water Absorption, Mechanical and Thermal Properties of Cellulose-High Density Polyethylene Composites

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In this study some physical, mechanical and thermal characteristics of high density polyethylene (HDPE) and $CaCO_3$ coated/pigmented wood free paper fiber composites were investigated. The fillers used in this study were uncoated cellulose, 5.8 %, 11.5 %, 16.5 % and 23.1 % $CaCO_3$ coated wood free paper fibers. Each filler type was mixed with HDPE at 40% by weight fiber loading. In this case, the ratio of $CaCO_3$ in plastic composites was calculated as 0 %, 2.3 %, 4.6 %, 6.6 % and 9.2 % respectively. Increased $CaCO_3$ ratio improved the moisture resistant, flexural and tensile strength of cellulose-HDPE composites. However, the density of the cellulose-HDPE composites increased with $CaCO_3$ addition. Energy Dispersive Spectroscopy on Scanning Electron Microscope analysis demonstrated the uniform distribution of $CaCO_3$ and cellulose fiber in plastic matrix. In addition, the thermal properties of fiber plastic composites were investigated. The results of Differential scanning calorimetry analysis revealed that the crystallinity of the samples decreased with increasing $CaCO_3$ content. Consequently, this work showed that $CaCO_3$ coated waste paper fibers could be used as reinforcing filler against water absorption in thermoplastic matrix.

Keywords: calcium carbonate, coated paper fiber, HDPE, plastic composite.

1. INTRODUCTION

Nowadays, because of environmental aspects and low costs the use of natural fibers as reinforcing filler to substitute synthetic fibers in composite material is being attractive. Wood, sawdust, paper mill sludge, agro-residue and waste paper are the good examples of the natural fibers utilized in thermoplastic composites [1 – 4]. Among these, waste paper is one of the largest recyclable components of the solid waste stream. Recovered paper constituted 54 % of total paper and board production in 2013 [5]. Recycled waste papers could be an ideal source as filler for plastic composites industries.

In the literature, there is some research about waste paper usage as filler in plastic composites. The mechanical and thermal properties of newspaper fiber reinforced rPET composites were examined by Ardekani and coworkers [6]. They concluded that SEBS-g-MA usage improve the impact strength but decrease the tensile and flexural strength of composites. Also they concluded that newspaper fiber addition in rPET increase the crystallinity and thermal stability of composites. Recycling disposable cups usage into paper plastic composites were investigated by Mitchell J and coworkers [7]. They improved mechanical properties of WPC with 40 wt.% disposable cup flakes addition into polypropylene.

Sailaja and Deephti studied mechanical and thermal properties of LDPE and esterified unbleached wood pulp [8]. They improved the mechanical properties of composite plastic with LDPE grafted maleic anhydride.

Faisal and Salmah investigated the mechanical and thermal properties of compatibilized waste office white paper (WOWP) filled low density polyethylene composites (LDPE) [4]. They concluded that the tensile strength and the Young's modulus of LDPE/WOWP composites increase with WOWP filler loading. Soucy and coworkers studied the potential of paper mill sludge usage for plastic composites [9]. Their results revealed that kraft (chemical) paper mill sludge most suitable for plastic composites production than chemi-thermo-mechanical and thermo-mechanical paper mill sludge. Nevertheless, in the literature, there is no research about coated or mineral filled waste paper usage how affects the strength properties and moisture resistance of fiber plastic composites.

As it is known, most of the writing and printing paper grade contains a significant amount of mineral filler such as calcium carbonate, talc, kaolin and titanium dioxide. They are used in order to improve the optical properties, printing quality, surface smoothness of the final product. In addition, filler usage decreases the cost of paper [10]. For example, woodfree uncoated (WFU) papers have a filler content of 5 % – 30 % with $CaCO_3$ and woodfree standard coated fine papers have a filler content of 30 % – 50 % with $CaCO_3$ or clay. In addition, copy papers have a filler content of 10 % – 25 % [11]. Due to the mineral filler content, it may be advantageous to use coated or pigmented paper fiber

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source as filler in thermoplastic composites. Natural fiber plastic composites are more durable and absorbs less moisture than timber. However, the moisture resistant of natural fiber plastic composites is not good enough for outdoors usage. The water absorption problem of natural fiber-plastic composites could be solved with mineral addition such as calcium carbonate, talc, wollastonite and silica. Mineral usage in wood or natural fiber plastic composites decrease the water absorption of plastic composites and increase the bending strength and heat distortion capacity of plastic composites [12 – 14]. Huuhilo and coworkers studied the impact of mineral filler to the moisture resistance of wood-plastic composites [15]. Their results revealed that mineral adding had a positive effect on the water absorption and thickness swelling of the studied samples. Wang and coworkers studied the tensile properties of nano-calcium carbonate impregnated bamboo fiber reinforced PP composites [16]. They concluded that nano-$CaCO_3$ impregnation of bamboo fiber had a positive effect on the tensile strength of bamboo-plastic composite.

Based on this information, in this study, uncoated cellulose and $CaCO_3$ coated wood free paper (bleached cellulose) fibers were investigated for reinforcement of high-density polyethylene.

2. MATERIALS AND METHODS

2.1. Materials

Fillers used in this study were pigment-free cellulose fibers (Kombassan paper mill, Konya, Turkey), copy papers (Ve-ge corporation, İzmir, Turkey) and coated poster papers (Mopak corporation, İzmir, Turkey). Thermoplastic used in this study was virgin high-density polyethylene (HDPE S 0464 had a melt flow index of 0.25 – 0.40 g/10 min (2.16 kg and 190 ºC, ASTM D1238) and a density of 0.959 – 0.963 g/cm^3, Petrokimya Holding Corporation, Aliaga, Izmir, Turkey). Maleated polyethylene (MAPE) was used as the coupling agent (Priex®12031. Addcomp Polymer Additive Solutions Inc., Nijverdal, The Netherlands), its melt flow index and density were 23 – 31 g/10 min (5.0 kg and 190 °C, ISO 1133) and 0.948 g/cm^3 respectively.

2.2. Fiber preparation

First, all paper and cellulose samples were divided into 3 x 3 cm pieces. Then repulped in a Micro-Maelström laboratory pulper. Pulping was carried out at 45 to 50 °C for 10 min without chemical addition. After the pulping, fibers had dried to oven dry at 103 ± 2 °C for 24 hour. Then dried fibers were granulated into flour form using a Willey mill. Next screened to 45 mesh size (354 μ) particles and dried to less than 1 % moisture.

2.3. Ash contents of fibers

Ash contents of papers and fibers were determined according to TAPPI-T 211 om-02 test method. Due to mineral and fine fiber loss during pulping, ash contents of fibers decreased after fiber preparation. The content of the ash in paper is directly related to the added pigment ($CaCO_3$). Table 1 shows the ash content of fibers before and after pulping.

2.4. Compounding and composite manufacturing

The experimental design of the study is presented in Table 2. During the manufacturing process, depending on the formulation, granulated HDPE and pulp fiber, were mixed with MAPE in a high intensity mixer to produce a homogeneous blend. Then, this blend was compounded in a laboratory scale co-rotating single-screw extruder (RONDOL 3212). The four-barrel temperature zones of the extruder were maintained at 160, 165, 170 and 175 ºC during the production and the rotor speed was set at 60 rpm. Extruded samples were collected, cooled, and granulated into pellets respectively. Finally, pellets were compressed into 5 × 150 × 160 mm size composites for 3 min at 170 °C.

Table 1. Ash contents of fibers before and after pulping

Paper grade	Sample code	Ash cont. of papers, %	Ash cont. of fibers after pulping, %
Pigment-free cellulose, 100 %	A	1.2	0.6
Pigment-free cellulose (50 %) + copy paper (50 %)	AB	7.3	5.8
Copy paper, 100%	B	14.1	11.5
Copy paper (50 %) + coated poster paper (50 %)	CB	21.3	16.5
Coated poster paper, 100 %	C	30.7	23.1

2.5. Dimensional stability

For the water absorption (*WA*) and thickness swelling (*TS*) tests 50 (L) × 50 (W) × 5 (T) mm specimens were prepared according to ASTM D 570. The water temperatures were 23 ± 2 °C. Ten replicate specimens were tested for each formulation. The FPC test specimens were immersed in water for 1, 7, 14, 21, 28, 35, 42, 49, 56, 63 and 70 day. The *WA* and *TS* values were calculated using the following equations:

$$WA\ (\%) = 100[(W2 - W1)/\ W1], \qquad (1)$$

where WA, $W1$ and $W2$ are the water absorption (%), weight of the dry specimen (g) and weight of the wet specimen (g), respectively.

$$TS\ (\%) = 100[(T2 - T1)/\ T1], \qquad (2)$$

where TS, $T1$ and $T2$ are the thickness swelling (%), thickness of the dry specimen (mm) and thickness of the wet specimen (mm), respectively.

2.6. Mechanical properties

Testing of the produced composites was conducted in a climate-controlled testing laboratory. Flexural and tensile properties of all waste paper FPCs were determined. The flexural tests were conducted in accordance with ASTM D 790.

Test samples were cut to the dimensions of 150 (L) × 13 (W) × 5 (T) mm. The span length of each specimen was 80 mm. Samples were tested on a Zwick 50 KN. The rate of crosshead motion was 2.0 mm/min, which was calculated according to ASTM D 790 standard. The tensile tests were conducted according to ASTM D 638.

Table 2. Experimental design of the study

Filler (cellulose fiber) in FPC	Sample code	Estimated $CaCO_3$ content in FPC, %	MAPE, %	HDPE in FPC, %
Pigment-free cellulose fiber, 40 %	A	0.0	3	57
Pigment-free cellulose fiber + copy paper fiber, 40 %	AB	2.3	3	57
Copy paper fiber, 40 %	B	4.6	3	57
Copy paper fiber + coated poster paper fiber, 40 %	CB	6.6	3	57
Coated poster paper fiber, 40 %	C	9.2	3	57

Tests were performed at a rate of 5.0 mm/min. Seven specimens of each formulation were tested.

2.7. Thermogravimetric (TGA) and differential scanning calorimetry (DSC) analyses

A Perkin Elmer TGA-6000 thermal analyser was used for thermogravimetric analysis (TGA) of the samples. The heating rate was 10 °C/min under nitrogen at a flow rate of 20 mL/min. Tests were performed in temperatures ranging from room temperature to 600 °C.

Thermal analysis of the WPC samples was carried out on a differential scanning calorimeter (Perkin Elmer Instruments DSC 8000). All DSC measurements were performed with 6 ± 0.2 mg powdered samples under a nitrogen atmosphere at a flow rate of 20 mL/min. The degree of crystallinity (X_c) of the HDPE component was determined using the following equation [17]:

$$X_c = (\Delta H_m / \Delta H_0) \cdot (100/W) \cdot 100\ \%, \qquad (3)$$

where ΔH_m and ΔH_0 are the heat of fusion of the composites and 100 % crystalline polypropylene, respectively, and W is the filler proportion. In this calculation, ΔH_0 was taken to be 293(J/g) [17].

2.8. Energy dispersive spectroscopy on scanning electron microscope (SEM-EDS)

A Carls Zeiss Evo LS-10 scanning electron microscope and Bruker QuantaxXFlash 6/100 energy dispersive spectroscopy analyser was used to study the fractured surface of the samples and uniform distribution of $CaCO_3$ in polymer matrix. To prepare the fractured surfaces, the samples were first dipped into liquid nitrogen and snapped in half. The samples were then mounted on the sample stub and sputtered with gold. During energy dispersive spectroscopy analysis, gold coating correction was applied on software.

2.9. Fourier transform infrared (FTIR) spectroscopy analysis

The Shimadzu IR Prestige-21 FTIR equipped with ATR was used to study the characterization of the fiber-HDPE composites. The spectra were recorded between 600 and 4000 cm^{-1}, with 16 scans per experiment and a resolution of 8 cm^{-1}. All the spectra for each group were transformed into absorbance spectra averaged before baseline correction and normalization.

2.10. Statistical analysis

All results (mechanical and decay) were evaluated with analysis of variance (ANOVA). All means were compared using the Duncan multiple comparison test (95% confidence interval) using a SPSS statistical program (SPSS 19. 2010).

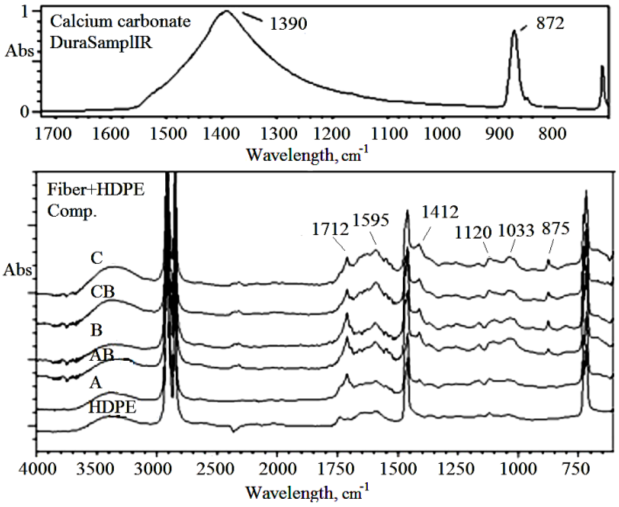

Fig. 1. FTIR spectra of fiber-plastic composites

3. RESULTS AND DISCUSSION

Fig. 1 shows the FTIR spectra of pure $CaCO_3$and fiber-plastic composites. The peak at 872 – 876 cm^{-1} band range was determined to characterize the content of $CaCO_3$ in fibers and FPCs according to pure $CaCO_3$ spectra and the studies of Feng [18] and Zhao et al [19]. The characteristic peaks of $CaCO_3$ at 872 cm^{-1} and 1390 cm^{-1} bands can be shown on pure $CaCO_3$. Depending on the $CaCO_3$ content in fiber-HDPE composites, the peak intensity at 875 cm^{-1} and 1412 cm^{-1} band were increased. FTIR results are consistent with ash content of fibers (Table 1). In addition, characteristic peaks of cellulose can be noticed at 1033 cm^{-1} and 1120 cm^{-1} band. The $CaCO_3$ content in plastic composites were corroborated with SEM-EDS results too. Chemical analysis by EDS show that Ca contents of plastic composites were 0.46 % for sample A (cellulose fiber-HDPE), 2.2 % for sample AB (copy paper fiber-cellulose-HDPE), 3.06 % for sample B (copy paper-HDPE), 7.27 % for sample CB (coated poster paper fiber-copy paper fiber-HDPE) and 8.91 % for sample C (coated poster paper fiber-HDPE). SEM images and EDS diagrams of fiber plastic composites are shown in Fig. 2 – Fig. 6. Fig. 7 and Fig. 8 show the water absorption and thickness swelling of samples respectively. As seen in figures, both water absorption and swelling happened during 70 days of sample immersion. However, the water absorptions of cellulose-plastic composites were diminished by increased $CaCO_3$ doses. Maximum thickness swelling (2.77 %) and water absorption (6.93 %) were obtained from ($CaCO_3$-free) cellulose fiber-HDPE composites after 70 days of immersion in water bath.

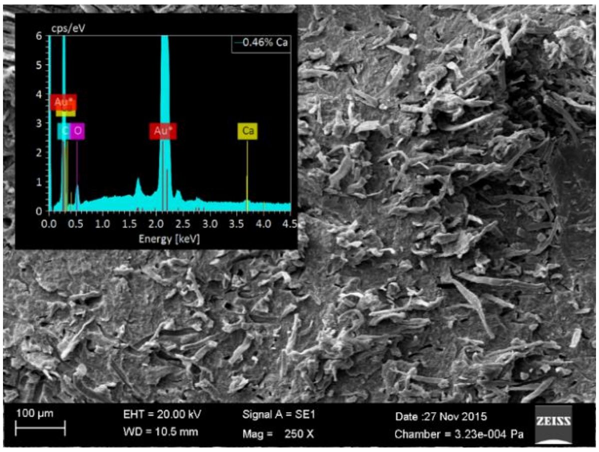

Fig. 2. SEM image and EDS spectra of sample A

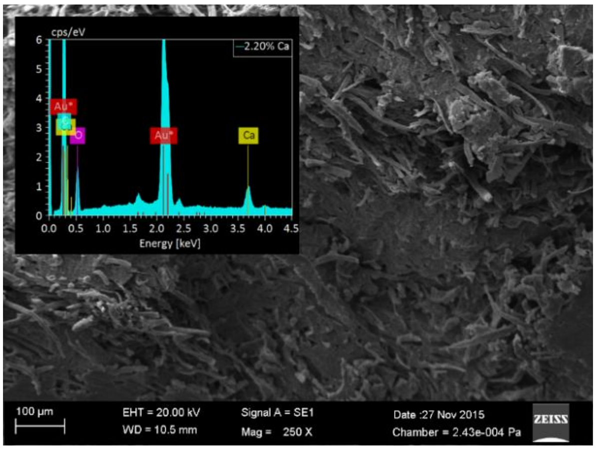

Fig. 3. SEM image and EDS spectra of sample AB

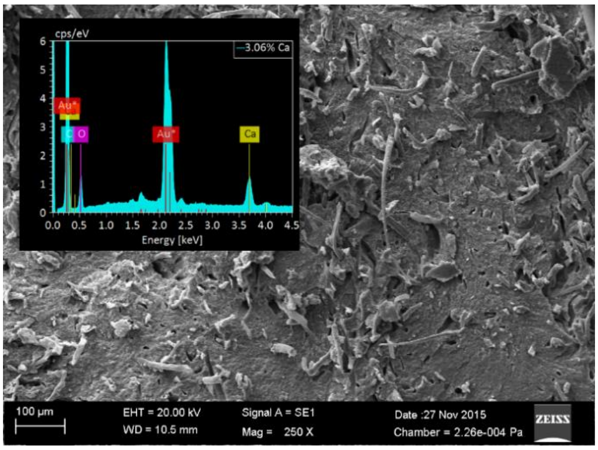

Fig. 4. SEM image and EDS spectra of sample B

After 14 days, thickness swelling was 2.27 % and water absorption was 5.34 % for the composites filled with cellulose fiber (sample A). The thickness swelling and the water absorption of composite decreased to 1.71 % and 3.67 % respectively after 14 days with 4.60 % $CaCO_3$ presence in cellulose-plastic composites. After 70 days, the thickness swelling was 2.00 % and the water absorption was 5.55 % for the 4.60 % $CaCO_3$ contained fiber-composite (sample B). When $CaCO_3$ presence increased to 9.2 % in cellulose-plastic composite, minimum thickness swelling and water absorption values were obtained. Similar results were observed by Huuhilo and coworkers [20].

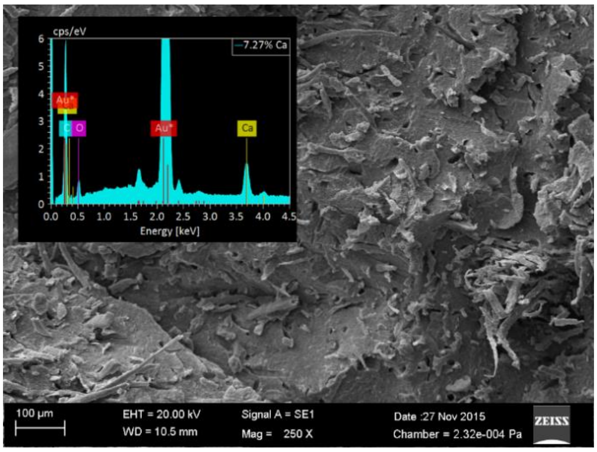

Fig. 5. SEM image and EDS spectra of sample CB

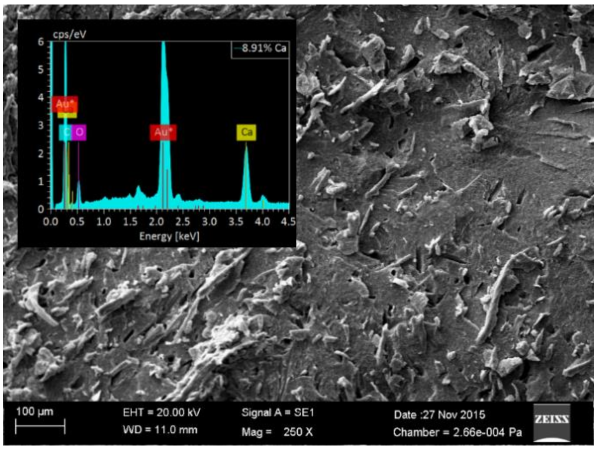

Fig. 6. SEM image and EDS spectra of sample C

Thickness swelling and water absorption of composite (sample C) was 1.09 % and 2.52 % after 14 days and 1.74 % and 4.29 % after 70 days respectively. The reason for the improvement in moisture resistance, could be explained by the filling of voids, formed between plastic and fibers, with $CaCO_3$. In addition, the density of fiber-HDPE composites increased from 0.97 kg/m^3 to 1.02 kg/m^3 with 9.2 % $CaCO_3$ charge.

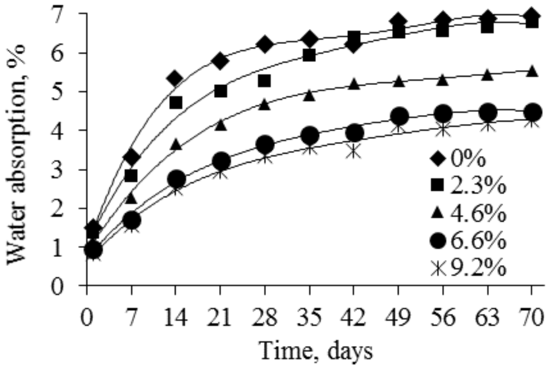

Fig. 7. Water absorption of samples

Tensile strength and flexural strength of fiber plastic composites are presented in Table 3. Tensile strength and flexural strength increased moderately with $CaCO_3$ presences but there is no significant difference between flexural and tensile strength values of composites ($p < 0.005$). Similar trends were observed at flexural and tensile modulus values of composites. Huuhilo and coworkers had acquired similar results for mineral reinforced wood-plastic composites [20].

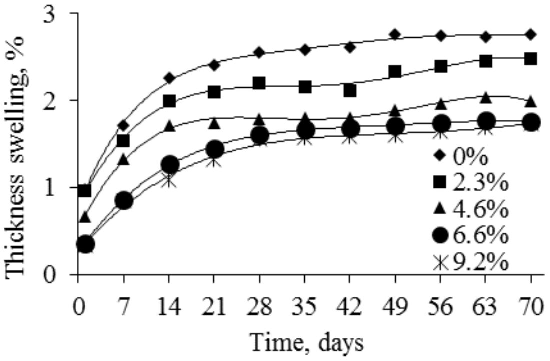

Fig. 8. Thickness swelling of samples

Table 3. Mechanical properties of fiber-HDPE composites

Sample code	Flexural strenght, MPa	Flexural modulus, GPa	Tensile strenght, MPa	Tensile modulus, GPa
A	20.92 *ab* (1.27)	2.46 *a* (0.34)	17.58 *a* (0.68)	1.93 *ab* (0.08)
AB	19.52 *a* (1.32)	2.46 *a* (0.15)	17.56 *a* (1.17)	1.86 *a* (0.30)
B	20.93 *ab* (1.80)	2.48 a (0.26)	17.59 *a* (1.23)	1.87 *a* (0.07)
BC	21.15 *b* (1.49)	2.62 *a* (0.30)	18.03 *a* (0.78)	2.06 *b* (0.08)
C	21.78 *b* (0.85)	2.72 *a* (0.16)	18.23 *a* (1.12)	2.03 *ab* (0.11)
*Note: The value in parentheses is the standard deviation; each value is the average of seven samples tested; groups with the same letters in the column indicate that there is no statistical difference ($p < 0.05$).				

The TGA data and derivatives of TGA data for fiber-HDPE composites are shown in Fig. 9 and Fig. 10 respectively. The first degradation peak was observed at a temperature of approximately 370 °C, which was due to the degradation of cellulose. The second degradation peak in the TGA curve was observed at 490 °C, caused by the degradation of HDPE matrix in the composites. The residual weight (%) of samples increased with increased doses of $CaCO_3$ after burning.

The DSC results of fiber-HDPE composites are given in Table 4. The observed melting temperatures and ΔH values were lower than for the pure HDPE. The increased doses of $CaCO_3$ in composites decreased the calculated degree of crystallinity (X_c %). Similarly, Zuhaira and Rahmah reported that the degree of crystallinity (X_c) of kenaf-HDPE hybride composite decrease with the addition of $CaCO_3$ filler loading [21].

Both the TGA and DSC analysis results indicated that $CaCO_3$ doses in fiber did not affect the thermal properties of fiber-HDPE composites significantly. Martikka and coworkers examined the effect of mineral fillers on the thermal properties of wood plastic composites and they had acquired similar results in their study [22].

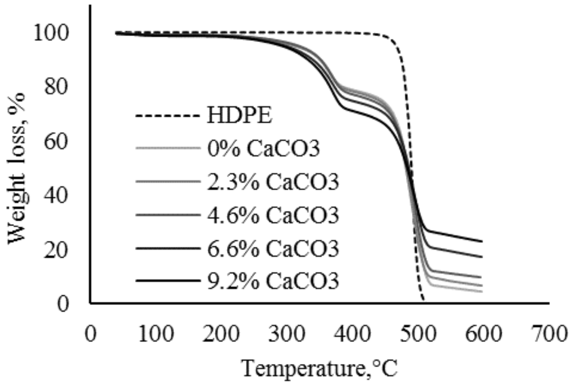

Fig. 9. TGA curve of samples

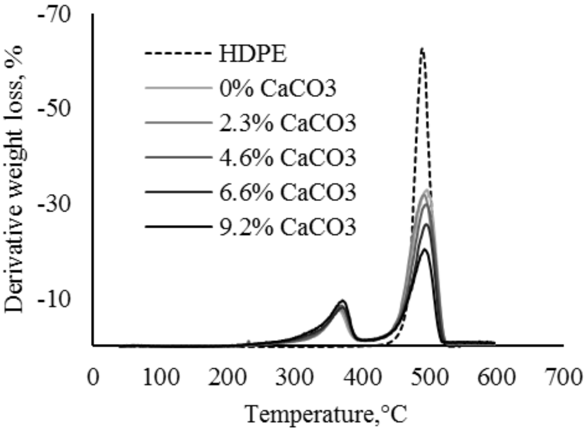

Fig. 10. Derivatives of TGA curve

4. CONCLUSIONS

In this study some physical, mechanical and thermal characteristics of high density polyethylene (HDPE) and $CaCO_3$ coated wood free paper fiber composites were examined. Calcium carbonate presence had a positive effect on the water absorption and thickness swelling of the cellulose fiber-HDPE composites. With 9.2 % $CaCO_3$ presence in cellulose fiber-HDPE composites, the water absorption decreased from 5.34 % to 2.52 % after 14 days immersion in water.

Table 4. DSC results of samples

Sample code	*T* cold, °C	Peak height cold, mW	Δ*H* cold, J/g	*T* melt, °C	Peak height melt, mW	Δ*H* melt, J/g	*X* corr, %
HDPE	117.69	-49.97	-171.93	133.95	31.69	171.95	60.16
A	116.82	-29.70	-135.09	130.64	28.42	138.83	69.39
AB	117.41	-18.71	-129.96	129.60	16.39	127.52	63.74
B	117.92	-23.63	-129.96	130.82	18.93	124.26	62.11
CB	117.90	-19.32	-122.55	130.66	16.46	120.20	60.08
C	117.55	-15.07	-76.64	129.22	13.26	119.90	59.93

In addition, the thickness swelling of composites decreased from 2.68 % to 1.09 % after 14 days immersion in water. Tensile strength and flexural strength increased moderately with $CaCO_3$ presences in composites. Both the TGA and DSC analysis results indicated that $CaCO_3$content in fiber did not affect the thermal properties of cellulose fiber-HDPE composites significantly. However, increased $CaCO_3$content decreased the calculated degree of crystallinity of composites slightly. The results of this study show that the moisture resistant of fiber plastic composite can be increased with coated waste paper fiber usage as filler.

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