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A study on effects of modified calcite on filler retention and mechanical properties of fluting papers

Modifiye kalsitin dolgu tutunumu ve fluting kağıtların mekanik özellikleri üzerine etkileri

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ABSTRACT

In this study, the use of modified calcite as a filler in fluting paper production was investigated and its effects on retention and some mechanical properties of the papers were determined. Ground calcium carbonate (GCC/calcite) and cationic starch were used for modification. Filler modification with alum effect of CPAM resulted in starch-calcite encapsulation. Produced modified calcite (MC) was used in fluting paper production at certain dosages as filler. Simultaneously, paper was made with unmodified calcite (UC), and the effects of modified calcite on the paper properties and filler retention were studied. The results of this study indicated that the use of 10% MC improved filler retention and provided approximately 23% more retention than the use of 10% UC. In addition, mechanical properties and air permeability values of MC-filled papers were higher than those of UC-filled papers. With the use of MC in fluting papers production, better filler retention was achieved, while the reduction in mechanical properties caused by the addition of filler can be minimized. Besides, impurities in white water can be minimized by reducing the filler dosage given during paper production.

Keywords: Calcite, Starch, Modification, Fluting paper, Retention, Mechanical properties

Introduction

Paper, which is one of the most produced and consumed substances in the world, and that the development in production and consumption in the countries affects other countries instantly, has an important strategic position. There is an increasing fiber deficit in the world and it is stated that cellulose investment is required in various sources and large pine forests are needed. However, high capital cost is required in cellulose production (Laftah and Wan Abdul Rahman, 2016).

Cellulose is not produced in Turkey and imported cellulose costs are overmuch (Cicekler and Tutus, 2021). Paper makers are seeking for alternative solutions to minimize cellulose-related costs, whether in paper or paper packaging. The fillers used to improve the paper properties are therefore of great importance. (Lourenço et al., 2019; Nikkhah Dafchahi et al., 2021; Tutus et al., 2020; Zhang et al., 2013). Since these non-fiber inorganic materials are much cheaper compared to fiber materials, they are highly preferred because they reduce production costs and provide faster drying compared to fiber raw materials, thus increasing production (Karademir et al., 2013; Lee et al., 2021).

Many industrial raw materials are evaluated in paper production and used for two different purposes as filling and coating. Some minerals are used only in filling or coating, while others can be used in both areas (Beazley, 1991). According to the type of paper produced, approximately 25% filling mineral is used. Fillers improve some paper properties such as opacity, whiteness, ink absorption, surface smoothness, dimensional stability, printing quality, softness and durability properties. Features required from an ideal filler; high whiteness, proper refractive index and grain distribution, high degree of retention, insolubility in water, or very little dissolution, low density, chemically reactive, low abrasion and cheap (Gigac et al., 1995; Kim et al., 2011; Tutus et al., 2018).

While the market share of calcite ($CaCO_3$) was less than 1% in the European paper industry 25 years ago, it now has more than 40% of the market. The use of calcite has increased in the past decade and has been approved in alkali papermaking process, especially in Europe (Hubbe and Gill, 2016; Shen et al., 2009). It has an important position in the filling and white pigment markets. Calcite can be prepared with lower cost, preserves the mechanical characteristics of the paper, provides to use more filler in paper

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structure and improves whiteness, opacity and air permeability of papers without losing the brightness (Shen et al., 2010; Tutus et al., 2020). Calcite filled papers can keep their whiteness longer than filler-free papers. Because it is used in alkaline environment, it provides an advantage in terms of preventing environmental pollution (Geng et al., 2021; Shen et al., 2009).

Loss of binding due to increased mineral fillers leads to retention problems, as the fillers are more difficult to retention and more retention aids is required; this leads to formation and printing problems such as sheet delamination, picking, linting, dusting (Cadotte et al., 2007; Yang et al., 2013; Yoon and Deng, 2006). Therefore, the filler content in paper is rarely limited to values higher than 30% (Hubbe and Gill, 2016; Shen et al., 2009). For a paper mill producing 1800 tons/day, if it was possible to increase the filler content of a paper with an average of 30% filler content by 5%, a significant amount of fiber (90 tons/day) gain could be achieved.

Quality papers imported from foreign markets are at higher price and these prices reduce purchasing power and limit the use of quality papers such as carbonless paper, printing paper, copy paper, special thin papers. Although enough industrial raw materials are available for quality paper production in Turkey, it is still expected to be implemented. The technology required for the use of industrial raw materials has not been provided yet in paper industry. High-cost strength-enhancing chemicals are used to achieve high strength properties, which increases production costs (Strand et al., 2017). Considering the waste paper and cellulose costs, the use of low-cost fillers has gained importance. Beside, fillers are not fibrous, so they can cause decreases in strength properties of the papers. Some authors have investigated the modification of filler particles by starch or its derivatives or by filler particle encapsulation with a starch gel. Both techniques are very effective in improving the mechanical properties of the paper, such as the tensile, burst, and tear indices, without sacrificing optical properties (Cao et al., 2011; Kuusisto and Maloney, 2016; Shen et al., 2009; Xie et al., 2019). The purpose of the current study was to increase the filler retention and to prevent the decrease in mechanical properties due to the increase in the filler rate.

1. Materials and methods

1.1. Materials

Calcite (D50=2,8 µm) as filler taken from OMYA Mining Inc. was modified and used in this study. Technical properties of the calcite were given in Table 1.

Table 1. *Calcite properties used in the study*

Calcite Properties	Values
45 mesh screen (ISO 787-7), %	0.01
Top cut (D98, Malvern Mastersizer 2000), µm	13.0
< 2 µm (Malvern Mastersizer 2000), %	37.1
Average (D50, Malvern Mastersizer 2000), µm	2.8
Brightness CIELAB (ISO 11664-4), L*, a*, b*	98.5, -0.03, 0.8
Brightness RY (C/2º, DIN 53163), Ry	96.2
Moisture content, %	0.1
$CaCO_3$, %	98
$MgCO_3$, %	1.7
F_e2O_3, %	0.05
HCl insoluble content, %	0.2

Starch, retention aids and cationic polyacrylamide (cPAM) were purchased from the market. Fluting papers were produced by recycling old corrugated cardboards (OCC).

1.2. Calcite modification

Modified calcite production was carried out with the preparation steps of starch and calcite shown in Figure 1. Calcite was transferred to a beaker with distilled water to be 8% dry matter and mixed continuously to provide homogeneous slurry at 50 ºC. The starch was cooked in 5% dry matter at 92 ºC and cooled to 72 ºC for 30 minutes. Prepared calcite and starch slurries (w/w) with adding 0.03% CPAM by weight of dry fiber were transferred into a reactor and mixed for ten minutes (Figure 1).

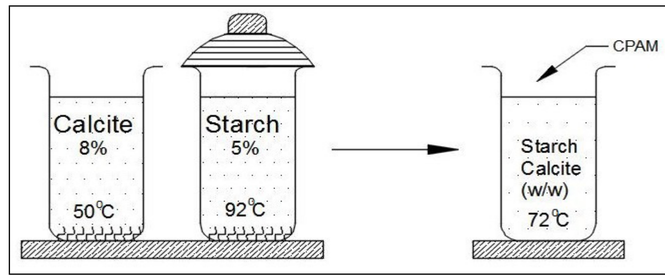


Figure 1. *Schematic view of calcite modification in this study.*

Starch-calcite encapsulation occurs in filler modification with alum effect of cPAM. In addition to the encapsulation of a single filler particle as shown in Figure 2, it is thought that it is possible to encapsulate several particles simultaneously to bind each other (Nelson and Deng, 2008; Shen et al., 2009; Yan et al., 2005; Zhao et al., 2008).

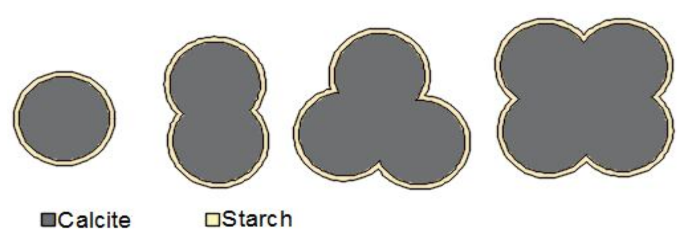


Figure 2. *Encapsulation structures of filler and starch*

1.3. Paper production and tests

Recycled OCC pulps were beaten to 40±2 Schopper-Riegler (ºSR) freeness level by using a Hollander beater. Beaten pulps were blended with modified (MC) and unmodified (UC) calcites and fluting papers with 90 grammages ($g.m^{-2}$) were produced with Rapid Kothen paper machine according to ISO 5269-2 standard. Starch (5%) was used only with UC filler in the paper production. Since calcite was modified with starch, no starch was added to the process with MC. Base papers (filler-free) were manufactured from the OCC pulps without the use of fillers in order to determine the effects of UC and MC on some mechanical properties.

The papers were incinerated in a crematorium at 575 ºC and resulting ash was weighed. The filler retention rates were calculated by the Equation 1 given below.

$$Retention\ Rate\ (\%) = \frac{Ash\ weight}{Paper\ weight\ (Oven-dried)} X\ 100 \quad (1)$$

After being conditioned for 24 hours in a conditioning room at 23±1 ºC and 50±2% relative humidity according to the TAPPI T402 standard, the fluting papers were subjected to tests according to relative standards given in Table 2.

Table 2. *The tests and standards applied to the fluting papers*

Tests	Standards
Breaking length (km)	TS EN ISO 13121
Burst strength (kg.cm^{-2})	TS EN ISO 2758
SCT (short span compression) (kN/m)	TS EN ISO 9895
CMT (corrugating medium test) (N)	TS EN ISO 9895
Scott bond (j.m^{-2})	TAPPI T569
Air permeability (s)	TAPPI T460
Filler rate (%)	TS EN ISO 1683

Ten fluting papers produced from each experiment were subjected to the tests specified in Table 2. To evaluate the effects of UC and MC on the fluting paper properties, the mean values of the properties were used. SEM images of produced papers were obtained using a ZEIZZ (Germany) microscope at an accelerating voltage of 0–30 kV.

2. Results and discussion

2.1. Findings on filler retention

Table 3 shows the measured filler doses and retention values in the manufacturing of fluting paper using UC and MC. The filler content of the base (filler-free) paper was determined as 10.4%. The filler rate is realized as 13.4% with using of 10% UC, while the increase in filler retention was 28.5%. Accordingly, the remaining part of the calcite was deposited onto the paper during sheet formation and the starch was effective for the filler retention. With the addition 10% MC during paper production, the filler retention increased about 58.7%.

Table 3. *Filler dosages and retention values in the fluting paper production using UC and MC*

Paper type	Filler Dosages (%)	Filler Retention (%)	Retention Increase (%)
Base paper	-	10.4	-
Paper filled UC	10*	13.4	28.5
Paper filled MC	5	14.2	36.4
	10	16.5	58.7
	15	21.4	105
	20	19.6	88.3

**Generally 10% filler is used in fluting paper production.*

When UC and MC were compared, it is clear that the use of MC significantly increases filler retention compared to UC (Figure 3). However, as the MC usage rate rises above 15%, its effect on the retention decreased from 105% to 88.3%. The advantages of using carbohydrate polymers in filler modifications can include low cost, ease of availability and environmental ease of modifiers, improved paper strength, enhanced filler retention or higher filling loading levels and successful marketing of the corresponding technology based on existing scientific papers (Fahmy and Mobarak, 2008; Fan et al., 2017; Fatehi et al., 2013; Shen et al., 2010).

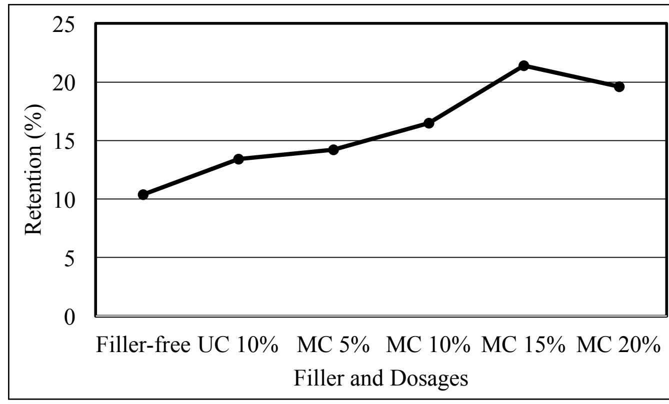


Figure 3. *Filler retention in the fluting paper production*

The amount of filler used in the system can be reduced if the filler particles are deposited into paper structure. Approximately 60% of the calcite filler used in paper production could be retained, with the remainder lost in circulation and cleaning water. By using less calcite in paper production, the recirculating water under the wire (white water) is less polluted. As a result, calcite damage in the treatment plant will be minimized by reducing the treatment load.

2.2. Mechanical properties of the fluting papers

The mechanical test results of the fluting papers containing no-filler, UC and MC were shown in Table 4. It is generally accepted that it has a negative effect on the strength-mechanical properties of papers with the increase of the filler content in the literature. Strength values of the papers used starch increased significantly compared to the filler-free paper due to the internal bonding property of starch.

Table 4. *Some mechanical properties of the fluting papers*

Papers	Filler Dosage (%)	SCT (kN.m^{-1})	Burst Strength (kg.cm^{-2})	CMT (N)	Scott Bond (j.m^{-2})	Breaking length (km)	Density (gr.cm^{-3})	Air permeability (s)
Filler-free	-	1.05	0.99	53.2	80.1	2.13	0.32	2.1
Starch	10	1.26	1.05	72.2	97.8	2.31	0.29	2.8
UC	10	1.20	1.00	65.0	89.5	2.04	0.29	2.6
MC	5	1.29	1.09	66.3	88.4	2.13	0.29	2.9
	10	1.33	1.19	68.9	85.6	2.12	0.31	3.3
	15	1.28	1.21	70.6	91.0	2.34	0.30	4.1
	20	1.44	1.31	72.0	97.8	2.45	0.29	5.4

In accordance with the general studies, the filler addition showed a significant decrease in values such as CMT and breaking length (Bajpai, 2018). Nevertheless, it is thought to be higher than the filler-free paper values, which is due to the internal bonding effect of starch. As the modified filler add-on increased, the generally expected strength values did not decrease, but an increase in non-significant values was observed (Figure 4). As the reason for the increases, it is seen that the effect of starch internal adhesion was realized, and with the increase in MC dosage, calcite grains encapsulated with cPAM support increased the retention by making wet-end bonding to the fibers and also preserved the strength (Chen et al., 2020; Mousavipazhouh et al., 2018) carboxymethyl cellulose (CMC.

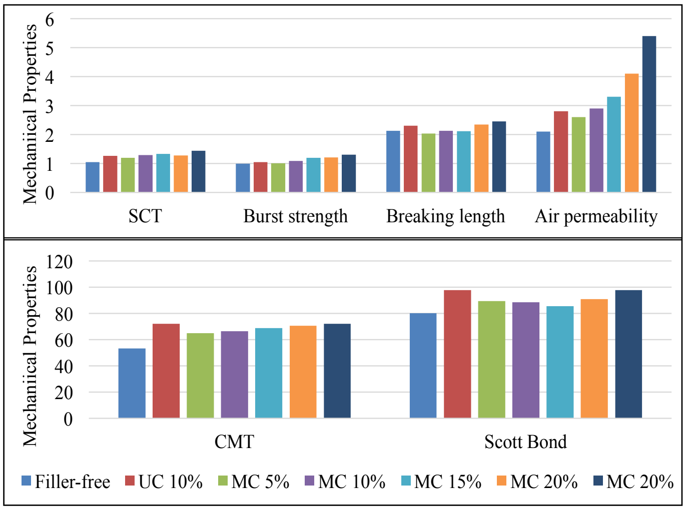
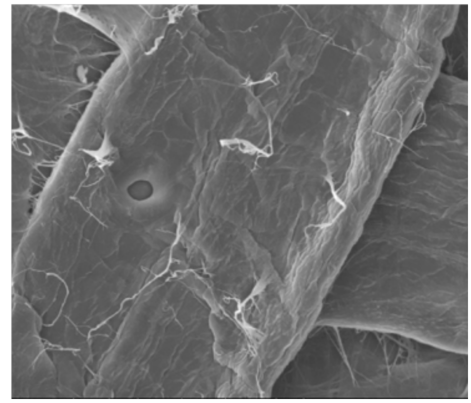


Figure 4. *The effects of the fillers on some fluting paper mechanical properties*

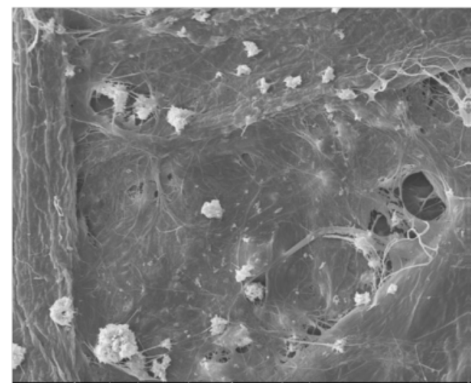
While the air permeability of the filler-free samples was measured as 2.1 s, the values increased as the MC dosage increases. Based on this, it can be said that the pores of the fluting paper were closed and the filler retention increased (Wu et al., 2016). SCT and CMT values of the fluting papers were increased by using 10% MC compared to 10% UC and filler-free papers. The respective values increased by 10.8% and 6% as a result of the use of MC. Filler addition often has effect of reducing stiffness (Hubbe and Gill, 2016). The CMT values are also high parallel to good paper stiffness values (Kiaei et al., 2016). The MC used in this study provided better stiffness than UC, and therefore the CMT values of MC-filled fluting papers were higher. Due to the buckling stability provided by short length tests compared to traditional test method, the material feature "compressive strength" can be measured with high accuracy using SCT. Starch use directly affects SCT, and an increase in the amount of starch used also positively affects SCT values (Andersson et al., 2013)therefore, is postulated to provide an added tool to support the necessary absorption control of the starch in respect to the packing density/permeability of the sheet as well as surface charge. To illustrate this behaviour, unfilled and filled laboratory-formed sheets without internal size are initially used, in which the starch is either cationically or anionically charged. The ground calcium carbonate (GCC. Scott Bond test is widely used method for determining paper and board delamination resistance. Scott Bond is a kind of indicator of inter fiber bond strength (Fellers et al., 2012). As mentioned before, the use of filler in paper production negatively affects the number of fiber-fiber bonds, and the MC used in this study minimized this negativity. As with other mechanical properties, breaking length and bursting strength of MC-filled fluting papers were better than UC-filled papers.

2.3. SEM images of the fluting papers

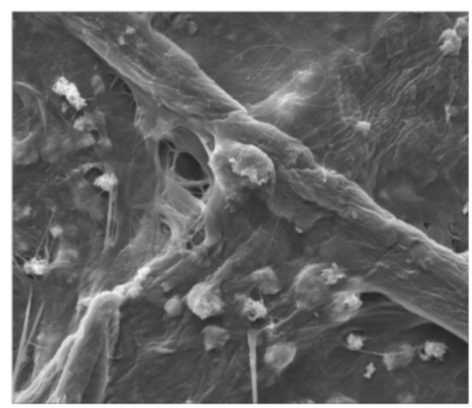
Figure 5 depicts scanning electron microscopy (SEM) images of the UC- and MC-filled fluting papers. It was discovered that the surface morphology of modified filler differed from that of unmodified filler, implying that surface encapsulation occurred during the modification process.



Filler-free (base) paper



UC-filled paper



MC-filled paper

Figure 5. *SEM images of the fluting papers (2000x)*

The morphology of the paper sheet was changed when fillers were included, and the filler particles stored in the fiber-based matrices onto the fiber surfaces. In compared to UC particles, the MC particles were noticeably more strongly and effectively attached to and bonded to the fiber surfaces.

Conclusion

The present study was designed to determine the effects of using MC as filler on filler retention and some mechanical properties of the fluting papers. The findings clearly indicate that the filler retention rate increased from 13.4% to 21.4% use of MC in fluting paper production. In this circumstance, the excess filler dosage given to the process could be decreased. Besides, the amount of calcite transferred to the white water could be decreased, and the load required for the treatment of the process water could be reduced. The results of this investigation show that the strength losses caused by filler addition of fluting papers production could also be minimized with use of MC. The breaking length (2.34 km) and burst index (1.21 kg.cm-2) of the MC-filled fluting papers were higher than those of filler-free fluting papers. The insights gained from this study may be of assistance to new researches about the calcite modification and its effects.

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