

New Diatomite Filler for Improvement of Paper Strength Properties

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Abstract: *In this work, diatomite particles were modified to improve the bondability of diatomite particles with pulp fibers and filler retention via a complex of cationic starch-sodium hexametaphosphate coating method. The particle size, FTIR spectroscopy characteristic and morphology of the resulting modified diatomite were studied to confirm the successful modification. The SEM results illustrated that the surface of modified diatomite particles was covered by the complex coatings. The coating efficiency of the complex of cationic starch-sodium hexametaphosphate on diatomite surface was up to 98%. Compared with the handsheet filled unmodified diatomite, the handsheet filled with modified diatomite had higher strength properties and lower bulk. When cationic starch dosage was 7%, and filler dosage was 25% , the resulting tensile strength of filled modified diatomite handsheet was 22.1% higher than the handsheet filled unmodified diatomite. Furthermore, with the filler loading of 30%, compared with that of unmodified diatomite, filler retention of modified diatomite was increased by 20.4%. The larger particle size and higher zeta potential of modified diatomite were beneficial to increase retention of modified diatomite filler.*

Keywords: *cationic starch, diatomite, filler modification, retention improvement, strength improvement*

1. Introduction

It has been a long history that mineral fillers are used in paper industry [1–3]. Many kinds of mineral fillers have been used, such as kaolin, calcium carbonate, diatomite, talcum powder and titanium dioxide [4–7]. Fibers are the most important component of paper. The second important component of the paper is mineral filler. The people always hope increase the filler ratio in paper to improve the cost competitiveness of paper products [8–12]. However, the use of filler has negative impact on the strength properties of the paper, especially at high filler addition level. One of the prominent problems in the use of filler is that many strength properties (such as tensile index , tear index) of paper were usually decreased with the increase of filler addition level [13–15]. Because of the above mentioned adverse effects, researchers have developed some techniques to overcome these shortcomings [13,16–20]. According to the different uses of paper, we can use a variety of methods to modify the filler (for example, chemical modification, physical modification and surface modification, etc.,) for acquiring the requirements of different paper and ultimately to improve the performance of the paper. [21–25]. In recent years, the surface modification of filler for improving the hydrogen bonding between fibers has been paying more and more attention [26–30].

Chemical structure of starch is similar to cellulose, and thereby starch is suitable for the surface modification of papermaking filler. Starch can be attached on the surface of filler to improve the bondability between fillers and fibers. It was found that adding modified filler can not only increase the retention rate of the filler, but also could increase the performance of the products while maintaining other indexes [31–36]. Diatomite is a kind of non-toxic, harmless and very environmentally friendly mineral material. Diatomite has been used in many fields. As a novel papermaking filler, diatomite has attracted more and more attention [37–40]. Compared with other fillers, diatomite has many excellent characteristics, such as small density, non-toxic, stable chemical properties, and more pores [41–55]. [12,56–59]. It is very meaningful and important to study whether can use cationic starch and sodium hexametaphosphate to modify diatomite for improving the strength properties of paper [60–67].

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In this work, diatomite particles were modified with cationic starch and sodium hexametaphosphate by coating method. The basic characteristics of modified diatomite particles including particle size, coating efficiency, FTIR spectroscopy characteristic, Zeta potential and surface morphology were evaluated. Meanwhile, the effect of modified diatomite filler on handsheet strength properties and filler retention was investigated.

2. Materials and methods

2.1. Materials

Diatomite was obtained from Hongyuan Co., Ltd. (Jilin, China). Cationic starch was purchased from Kang pu hui wei Co., Ltd. (Beijin, China). Sodium hexametaphosphate (SHP) was obtained from Aladdin Co., Ltd. (Shanghai, China). CPAM (Cationic polyacrylamide) was obtained from BASF Co., Ltd (China). APMP pulp board was obtained from a mill (Mudanjiang, China). The pulp was beaten in a Valley beater and the pulp was beaten to 45 °SR for use.

2.2 Methods

2.2.1. Preparation of Cationic Starch-Sodium Hexametaphosphate Complex Modified Diatomite

Diatomite particles were coated with cationic starch-sodium hexametaphosphate. This is a method of coating modification. The specific modification process was as follows: 3wt% cationic starch (the degree of substitution of cationic starch was 0.05) suspension was prepared, the cationic starch suspension was cooked at 90 °C for 1 h. Dry diatomite particles (10×10⁴ mg) were added to a 1L beaker, and then a certain amount of deionized water was added to the beaker, to form the final concentration of diatomite slurry was 15×10⁴ mg/L. The diatomite slurry was stirred by mixing with a stirrer for 10 min, and then the diatomite slurry was added into the above cationic starch suspension and stirred for a quarter of an hour to form the mixture. Sequentially, 3wt% sodium hexametaphosphate suspension was putted into the above mixture and stirred for a quarter of an hour and stood at 20 °C for two days. The supernatant of the above mixture was collected to measure the total organic carbon (TOC), and the sediment was washed by water until neutral. Finally, the sediment was dried at 85 °C for about 3 h. The cationic starch-sodium hexametaphosphate modified diatomite was gotten. The complex of cationic starch-sodium hexametaphosphate modified diatomite samples with different cationic starch/diatomite ratios were obtained by the above preparation procedure [68-72].

2.2.2. Characterization of Modified Diatomite Filler

The morphology characteristics of modified diatomite and unmodified diatomite were observed by SEM (SM6510A). The coating efficiency (CTEY, %) of the complex of starch-fatty acid on diatomite surface was evaluated by the content of the total organic carbon (TOC) in the supernatant evaluated by a TOC analyzer (Liqui TOC). CTEY was calculated by the following formula:

$$CTEY(\%) = [(W_i - W_d) / W_i] \times 100 \quad (a)$$

where, W_i and W_d (g) are the weights of the initial cationic starch-sodium hexametaphosphate complex and the cationic starch-sodium hexametaphosphate complex dissolved in the supernatant, respectively.

Malvern Zetasizer-3000 was used to measure the Zeta potential of modified diatomite and unmodified diatomite filler. OMEC LS-POP particle size analyzer was used to detect the particle size of diatomite filler. Using IRAffinity-1 FTIR spectrophotometer to test the FTIR spectrum of diatomite filler [72-80].

2.2.3. Handsheets Preparation and Test of Physical Properties

Bleached hardwood kraft pulp suspension (1%) were prepared, and then various dosages of diatomite filler were added into the pulp suspension and 0.5 wt% of cationic polyacrylamide were also added into the pulp suspension, let the mixer continue to stir until the mixture is absolutely mixed well. The handsheet was prepared with a Rapid-Koethen handsheet former. Grammage of the handsheets

were 60 g·m⁻². All handsheets were prepared at 105°C drying temperature, 1.0MPa pressure and the dried time was 10 minutes. The handsheets were then conditioned for 24 h (50±2% relative humidity, 23±1 °C). Physical properties of the handsheet was tested according to TAPPI Test Methods.

2.2.4. Measure of Filler Retention in Handsheets

The filler content in handsheets was tested by ashing in a muffle furnace (575±25 °C, 6 h). The filler retention (*FR*, %) was measured. The formula as follow (eq b):

$$FR (\%) = [M_1 / (D \times M_2)] \times 100 \quad (b)$$

where, M_1 (g) is the weight of ash in handsheets, and M_2 (g) is the weight of the filler added. D is the weight fraction of diatomite in filler.

Error Analysis: All the data were tested multiple measurements (3-8 runs) and a average value was reported. The error margin as follows: particle size(±3.5%), Zeta potential(±0.6%), filler retention(±0.5%), physical properties(±6%).

3. Results and discussions

3.1. Forming Mechanism and Characterization of Modified Diatomite

In the process of modified diatomite, researchers pay more and more attention to modifier renewable and environmental protection. Therefore, in this research, we chose the renewable of cationic starch and sodium hexametaphosphate as the modifying agent to coat surface of diatomite particles, because of cationic starch molecules contain a lot of hydroxyl groups. The hydroxyl groups can be form a hydrogen bond between the fibers. The schematic diagram was shown in **Figure 1**.

From figure 1, it could seen the whole modification process. Firstly, cationic starch adsorbed to the diatomite particles surface by adsorption. Secondly, diatomite particles had adsorbed cationic starch interact with sodium hexametaphosphate, because of the cationic starch and sodium hexametaphosphate can form complex (CSSC). Finally, diatomite particles were coated by CSSC. It is worth noting that in the whole process of modification, the control of various technological parameters is particularly important, such as cationic starch dosage, sodium hexametaphosphate dosage and surface charge of modified diatomite. These parameters directly determine the modification effect.

To understand the modification mechanism, we need to fully understand the material properties of diatomite. The surface of the diatomite particles also have a lot of hydroxyl groups, but because of its chemical inert itself. It makes difficult to react with fiber surface hydroxyl hydrogen and form the hydrogen bond. Therefore, we through the introduction of cationic starch in diatomite particles surface to increase the number of hydroxyl groups. The structure of cationic starch and cellulose are similar, so it is easy to form hydrogen bond between the hydroxyl. Therefore, the binding ability between diatomite particles and fibers can be improved.

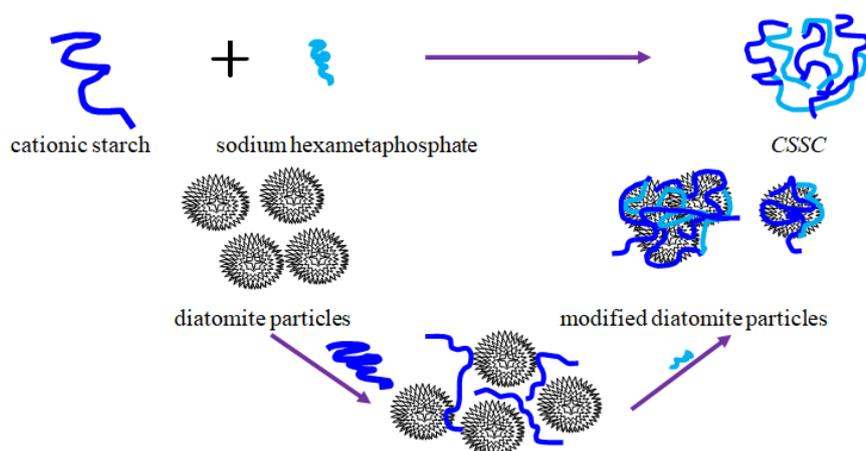


Figure 1. Preparation of modified diatomite using cationic starch and sodium hexametaphosphate

The SEM image of unmodified diatomite particles is shown in Figure 2a and the SEM image of modified diatomite particles is shown in Figure 2b. It was found that a significant difference between Figures 2a and 2b. It was also explained that diatomite particles were successfully coated with cationic starch-sodium hexametaphosphate complex. It is shown in Figure 2a that the image of unmodified diatomite retained its original morphology. From the Figure 2b, we can see that the modified diatomite particles were coated with a layer of complex. The surface of modified diatomite was fully covered and the micropores were invisible, which illustrated that diatomite was modified by the cationic starch-sodium hexametaphosphate complex. Since cationic starch has many hydroxyl groups in the molecular chain, the bondability of modified diatomite particles with fibers was enhanced. SEM images of modified diatomite particles show that the combination of cationic starch and sodium hexametaphosphate can effectively modify diatomite and make diatomite particles aggregate (Figure 2b), possibly due to the bridging function of the cationic starch-sodium hexametaphosphate complex. This aggregation phenomenon was beneficial to improve filler retention because of the larger size of composites/aggregates compared with the unmodified diatomite particles.

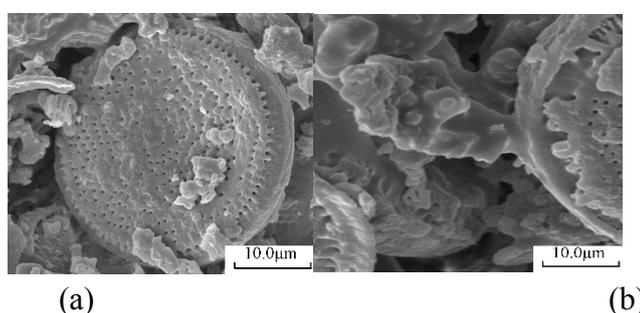


Figure 2. SEM images of unmodified (a) and modified (b) diatomite fillers.

For modified diatomite, it is very important to effectively utilize cationic starch-sodium hexametaphosphate complex. If the utilization efficiency of cationic starch-sodium hexametaphosphate complex is low, a good coating could not be formed on the surface of diatomite. In this research, the *CTEY* of cationic starch-sodium hexametaphosphate complex on the surface of diatomite was measured, and the results are shown in Table 1.

Table 1. *CTEY* of cationic starch-sodium hexametaphosphate complex on the surface of diatomite.

sample no.	Sodium hexametaphosphate dosage (%)	<i>CTEY</i> (%)
1	3	98.13
2	4	98.17
3	5	98.29
4	6	98.40
5	7	98.43

It was found that the *CTEY* of cationic starch-sodium hexametaphosphate complex on the surface of diatomite were all more than 98%. It indicated that cationic starch-sodium hexametaphosphate complex could be effectively coated on the surface of diatomite under the experimental conditions. Sodium hexametaphosphate dosage had almost no effect on *CTEY*, which illustrated that 3% sodium hexametaphosphate was enough.

The zeta potential of diatomite particles was tested, in order to estimate the change of diatomite particles charge and observe the impact the filler retention and flocculation of diatomite filler on fibers. It is important a factor that the Zeta potential of diatomite particles are affect application performance of diatomite filler in pulp fibers networks. In this work, the Zeta potentials of unmodified and cationic starch-sodium hexametaphosphate complex modified diatomite fillers with different dosages of

cationic starch were measured. Table 2 illustrates the influence of cationic starch dosage on the zeta potential of diatomite particles.

Table 2. Zeta potential of cationic starch-sodium hexametaphosphate complex modified diatomite filler

sample no.	cationic starch dosage (%)	Zeta potential (mV)
1	0 (unmodified diatomite)	-63.12
2	3	-25.31
3	5	-15.22
4	7	1.29
5	9	10.21
6	11	13.12

The electropositive of modified diatomite particles were enhanced compared with unmodified diatomite particles (-63.12 mV). Furthermore, the Zeta potential enhanced with the enhance cationic starch use level. The low cationic starch dosage modified diatomite particles were less positively charged than that of the other cationic starch dosage. The Zeta potential of modified diatomite particles had become to positive when the cationic starch dosage was 7%. The Zeta potential of modified diatomite was 1.29mV. It is primarily because that cationic starch can contribute significantly to improve the Zeta potential of the modified diatomite. Consequently, the introduction of cationic starch-sodium hexametaphosphate complex coated the diatomite particles surface, it is advantageous to increase the Zeta potential.

The particle sizes of unmodified and modified diatomite are both presented in Figure 3. Filler particle size is very important because it affects paper physical properties and filler retention. From Figure 3, it could seen that the unmodified diatomite average particle size was 4.81 μm . The modified diatomite average particle size was larger than unmodified diatomite. the modified diatomite average particle size was significantly increased to 6.28–7.61 μm after modification. The particle size of modified diatomite became larger compared with unmodified diatomite particles. It was probably because that cationic starch-sodium hexametaphosphate complex caused multiple diatomite particles to clump together. The process of coating and aggregation of diatomite particles was shown in Figure 1. The cationic starch-sodium hexametaphosphate was able to coat one single diatomite particle or several diatomite particles. The increase of Zeta potential and particle size of diatomite was helpful to improve the performance of diatomite.

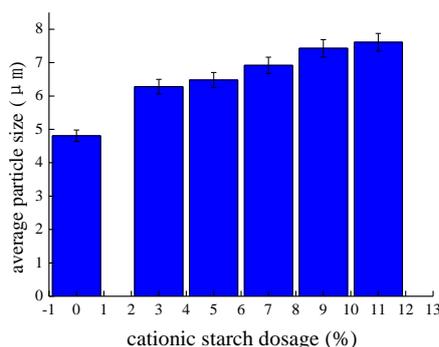


Figure 3. Effect of cationic starch on the particle size of diatomite

As Figure 4 shown, the characteristic peak at 1082 cm^{-1} is attributed to Si-O stretching vibration and the characteristic peak is the main characteristic peak of unmodified diatomite. The peak at 751 cm^{-1} is belong to Si-O-Si stretching vibration. The peak at 510 cm^{-1} is attributed to Si-O-Si. Compared with unmodified diatomite, modified diatomite shows a lot of new characteristic peaks. New characteristic peak appears at 3250 cm^{-1} belonging to the OH characteristic peak. The peak at 1075.8

cm^{-1} is attributed to C6-O-H stretching vibration of cationic starch molecular chain. This indicated that cationic starch had incorporated into diatomite. The peaks at 1151.7 cm^{-1} is P=O vibration peak and the peaks at 1284.6 cm^{-1} is P-O-C vibration peak. These characteristic peaks proved that cationic starch and sodium hexametaphosphate had been crosslinked, and the starch-sodium hexametaphosphate complex had coated on the surface of diatomite particles. It was also proved that the cationic starch basic chemical structure did not damage. So can provides the guarantee for the modified diatomite application in papermaking.

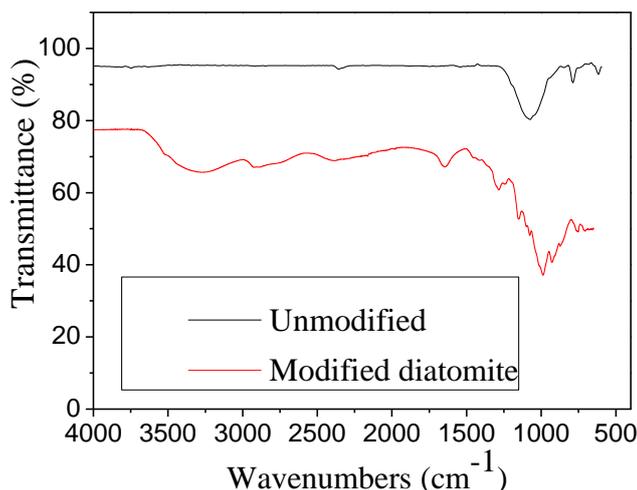


Figure 4. FTIR spectra of unmodified and modified diatomite fillers

3.2. Impact of Modified Diatomite on Filler Retention

The filler retention rate was a very important index in papermaking. The cationic starch-sodium hexametaphosphate modified diatomite fillers retention with different dosages of cationic starch were measured. The effect of cationic starch to the filler retention is shown in Figure 5a. The fillers retention of modified diatomite increased with increasing cationic starch dosage (the filler dosage was 30%). The modified diatomite retention was higher than 88% when cationic starch dosage was 7%. However, The fillers retention of modified diatomite increased slightly although the cationic starch dosage continued to improve when cationic starch dosage was over 7%.

Figure 5b illustrates that the filler retention rate of modified diatomite filler (cationic starch dosage was 7%) was higher than unmodified diatomite filler. The filler retention rate of the unmodified diatomite filled handsheet was lower than that of the modified diatomite filled handsheet. At a filler loading of 30%, the modified diatomite retention was higher than 88%. However, the retention rate of unmodified diatomite filler was under 74%. In this case, the cationic starch-sodium hexametaphosphate modification was beneficial to increase filler retention. It was mainly because that the cationic starch-sodium hexametaphosphate coated diatomite particles formed small aggregates (6.28-7.21 μm) rather than individual particles. Moreover, the zeta potential of modified diatomite particles were increased, which was beneficial to enhancing the retention. The increased zeta potential by adding cationic retention agent, the zeta potential of diatomite filler surface can be improved, and the retention of filler between fibers can be improved.

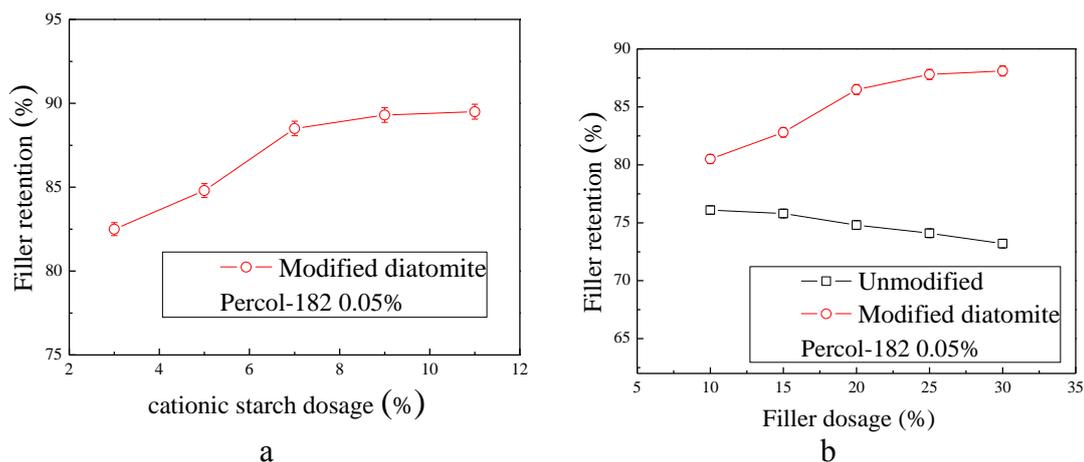


Figure 5. Effect of cationic starch dosage on filler retention(a) and filler dosage on the filler retention (b)

3.3. Strength Properties of Modified Diatomite Filled Handsheet

As shown in Figure 6. It is illustrated that the effect of the modified diatomite dosage and unmodified diatomite dosage on physical properties. The results shown that the strength properties of the handsheets became lower and lower when the dosage of fillers increased. It can be seen that the strength properties of the handsheets filled with unmodified diatomite decreased more and faster. It was mainly because that the dosage of unmodified diatomite filler increased which led to the decrease of the bonding opportunities between the fibers. However, the unmodified diatomite filler particles couldn't effectively combine with the fibers. In addition, when the modified diatomite filler dosage was increased, the modified diatomite itself will be scattered unevenly. Fiber compound together by bridge connection of modified diatomite filler gets decreased.

The connection opportunity of the fibers each other gets decreased. However, the strength properties of the handsheets using modified diatomite were higher when the same filler content. Especially for high dosage cationic starch as modifier, with the filler dosage improved, the tensile strength of modified diatomite filled handsheets increased by 19 and 69% compared with unmodified diatomite filled paper when the dosage of diatomite filler was improved from 10 to 30%. It is mainly because that the modified diatomite was coated with cationic starch-sodium hexametaphosphate complex. The modified diatomite has rich hydroxy groups. Therefore, the hydroxyl groups on the surface of modified diatomite particles could form hydrogen bonds with the fibers. Moreover, filler and pulp fiber would be more tightly bound due to the coating of cationic starch-sodium hexametaphosphate complex. Therefore, the tensile strength of the handsheet filled with modified diatomite was effectively improved.

As shown in Figure 6, the bulk of handsheet was also investigated. The bulk of handsheet increased with the increase of filler addition level. Meanwhile, the bulk of modified diatomite filled handsheet was lower than that of unmodified diatomite filled handsheet. It indicated that the cationic starch-sodium hexametaphosphate complex coatings on the surface of diatomite could improve the bondability of diatomite particles with pulp fibers. All the above results indicated that cationic starch-sodium hexametaphosphate complex modified diatomite can improve the strength properties of filled handsheet.

Nowadays, there are more and more diatomite modification methods. In this work, diatomite particles were modified with cationic starch and sodium hexametaphosphate by coating method. Compared with the unmodified diatomite, the modified diatomite can effectively improve the filler retention, strength properties of modified diatomite filled handsheet [70-85].

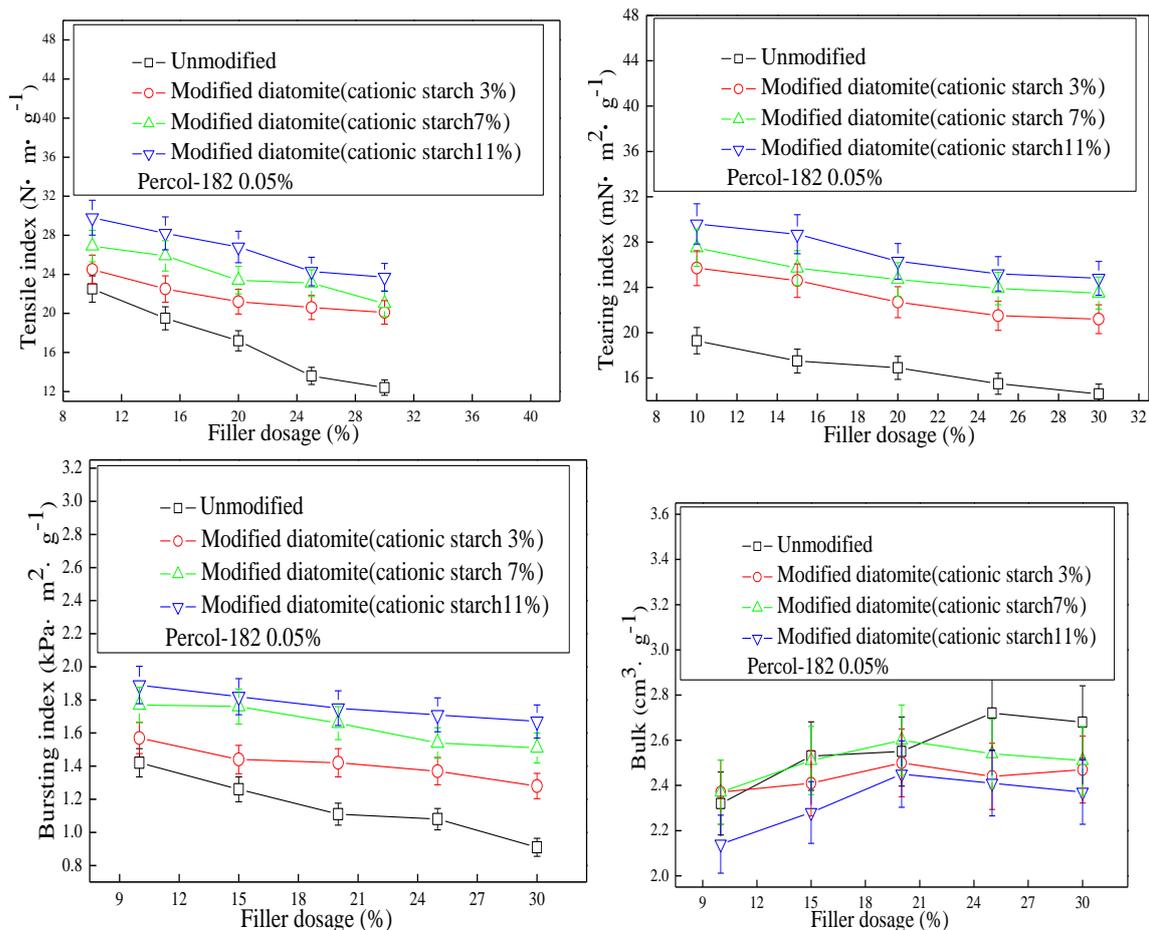


Figure 6. Effect of filler dosage on physical properties of filled handsheet

4. Conclusions

In this study, cationic starch and sodium hexametaphosphate were proved to be effective modifiers in the modification of diatomite. The utilization efficiency of cationic starch-sodium hexametaphosphate complex is high a good coating could be formed on the surface of diatomite. The coating efficiency (CTEC) was high as 98%. The SEM observations and particle size analysis confirmed the formation of modified diatomite filler. Compared with unmodified diatomite, Zeta potential of modified diatomite could be effectively raised, and it could change to electropositive. Modified diatomite effectively enhanced the retention of the diatomite filler. The physical properties of paper were significantly improved. As to high dosage cationic starch unmodified diatomite filled paper, at the filler content of 25%, the tensile strength is 22.1% lower than that of modified diatomite filler paper.

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