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Research Article

Processing and Characterization of Sepiolite Clay Containing Composites for Organic Brake Pad Application

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Abstract

Brake friction materials are crucial in transportation means. Organic brake pads are widely used in railway or automotive especially due to their low manufacturing cost compared to semi-metallic or ceramic pads. Owing to the complex requirements of brake lining systems, organic composite brake pads are a mixture of various components and are basically composed of binders, friction modifiers, fibers, lubricants, abrasives and various fillers. The choice of functional fillers and the optimization of the formulation is essential for improving the performances of these materials. Studies on the use of clay minerals such as kaolin or montmorillonite show that these fillers are effective. Therefore, in this study, a clay mineral, sepiolite was chosen. The composite samples prepared in this work are composed of Novolac resin as binder, cashew nut shell oil, ceramic fibers, glass fibers and other fillers. This study aims to explore the effect of sepiolite clay on the processing and on properties such as the density, the porosity or the hardness of the samples. Therefore, composite samples containing different amounts of sepiolite clay (5 wt. %, 10 wt. %, 15 wt. % and 20 wt. %) were prepared. For this purpose, the different components were mixed in a high speed mixer and pressed in a hot press at 160°C. Then, a post curing step at 180°C was applied. The density of the samples was determined and decreases from 2.22 g/cm^3 to 2.10 g/cm^3 with increasing sepiolite clay content. The porosity decreases from 4.47 % to 2.05% with increasing clay content until 15% and rises again to 2.81% with 20% of sepiolite content. Shore D hardness follows an inverse tendency to the density and rises from 85.3 to 90.8.

Keywords: brake pads, composites, sepiolite.

Organik Fren Balatası Uygulamalarına Yönelik Sepiyolit Kili İçeren Kompozit Üretimi ve Karakterizasyonu

Öz

Ulaşım araçlarında fren sürtünme malzemeleri büyük önem arz etmektedir. Yarı metalik ve seramik fren balatalarına kıyasla özellikle düşük üretim maliyetlerine sahip olduklarından dolayı, organik fren balataları demiryollarında ve otomotiv sektöründe yaygın olarak kullanılmaktadır. Fren balataları, karşılamaları gereken çok sayıda ve karmaşık gereksinimlerden/özelliklerden dolayı çok sayıda bileşenden oluşmaktadır. Bu karmaşık sistemler temel olarak bağlayıcılar, sürtünme düzenleyiciler, fiberler, yağlayıcılar, aşındırıcılar ve çeşitli dolgu malzemelerinin karışımından meydana gelmektedir. Fonksiyonel katkı malzemelerinin seçimi ve formülasyonun optimize edilmesi bu malzemelerin performansının iyileştirilmesinde başlıca rolu oynar. Yapılan çalışmalar, kaolin, montmorillonit gibi kil minerallerinin kullanımının balataların özelliklerinin iyileştirilmesinde etkili olduğunu göstermektedir. Bu nedenle bu çalışma kapsamında bir kil minerali olan sepiyolit seçilmiştir. Bu çalışmada novolak reçine (bağlayıcı olarak), kaju kabuğu yağı, seramik fiber, cam fiber ve diğer dolgu malzemelerinden oluşan numuneler hazırlanmıştır. Bu çalışmanın amacı sepiyolit kilinin kompozit numunelerin yoğunluğu, porozitesi ve sertliği gibi özellikleri üzerine olan etkisini araştırmaktır. Bu doğrultuda farklı miktarlarda sepiyolit kili içeren kompozit numuneler hazırlanmıştır (5 wt. %, 10 wt. %, 15 wt. % ve 20 wt. %). Kompozit numunelerin hazırlanma aşamasında bileşenler (Novolak reçine, kaju kabuğu yağı, seramik fiber, cam fiber ve diğer dolgu malzemeleri) yüksek hızlı karıştırıcı ile karıştırılmış ve ardından 160°C de preslenmiştir. Numunelere daha sonra 180°C de post kürleme işlemi uygulanmıştır. Numunelerin yoğunluğu ölçülmüş ve sepiyolit miktarı arttıkça 2,22 g/cm^3'den 2,10 g/cm^3'e düştüğü belirlenmiştir. Ayrıca sonuçlar göstermektedir ki porozite %15 sepiyolit oranına kadar %4,47'den %2,05'ye düşmüştür. Bunun yanı sıra %20 sepiyolit oranında porozite tekrar %2,81'e yükselmiştir. Numunelerin Shore D sertliği ise yoğunluğun aksine sepiyolit oranı arttıkça 85,3'den 90,8'e yükseliş göstermektedir.

Anahtar Kelimeler: fren balataları, kompozitler, sepiyolit.

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1. Introduction

Composite brake pads are important for human safety in passenger transportation. Organic composite brake pads are composed of a novolac phenolic resin filled with a wide range of inorganic fillers. Those materials are classified as friction materials a safety factor must be considered during their development. Therefore, most of the performance criterion of friction product related to safety (Ali & Joshi, 2015). Friction materials generally may contain more than 30 components that can be classified into four classes, namely fibrous reinforcement (Gopal, Dharani, & Blum, 1994), binders (Shin, Cho, Lee, & Jang, 2010), filler and friction modifier (Lee, Hwang, Lee, Cho, & Jang, 2010), (Han, Tian, & Yin, 2008). Each component is crucial and has an important influence on the properties of the final material. Frictional properties but also strength, stiffness or thermal stability are improved using fibrous reinforcements. Due to their low cost, mineral fillers are used and their principal role is to increase the friction lining volume which reduces the general cost. Barytes and clays are often used as fillers in friction materials (Morshed & Haseeb, 2004).

The choice of functional fillers and the optimization of the formulation is essential for improving the performances of this materials. Studies on the use of clay minerals such as kaolin or montmorillonite show that these fillers are effective. Therefore, sepiolite (SP) is a good filler candidate for composite brake pads. This natural clay mineral with a fibrous structure is composed of magnesium hydrosilicate and it belongs to the sepiolite - paligorskite group. Sepiolite is also called as attapulgite (Vicente Rodriguez, Lopez Gonzalez, & Banares Munoz, 1994). Sepiolite exists in nature in two types: the hydrothermal type α-sepiolite and the sedimentary type β-sepiolite. The latter has a fibrous structure similar to asbestos (Alvarez, 1984), (Hou, Ouyang, Zheng, Zhang, & Yang, 2017). Its use as an alternative to asbestos in brake composite was investigated by Hou et al. in 2017 (Hou, Ouyang, Zheng, Zhang, & Yang, 2017). The use of attapulgite ultra short fibers as an alternative for asbetos was also investigated by Washabaugh in 1986 (Washabaugh, 1986) ; (Ayar, 1994). Moreover, Boz and Kurt (Boz & Kurt, 2006) investigated the effect of sepiolite on bronze based friction materials.

Therefore, in this study the sepiolite clay containing composites for organic brake pad application were prepared. The composite samples are composed of Novolac resin as binder, cashew nut shell oil, ceramic fibers, glass fibers and other fillers. The aim of this study is to investigate the effect of sepiolite clay on the processing and on properties such as the density, the porosity or the hardness of the samples. Therefore, composite samples containing different amou%nts of sepiolite clay (5 wt. %, 10 wt. %, 15 wt. % and 20 wt. %) were prepared. Sepiolite clay was associated to a composite formulation developed by Metisafe. Properties such as density, porosity and Shore D hardness were characterized.

2. Materials and Method

2.1. Materials

Novolac type phenolic resin and hexamethylenetetramine (HMTA) were supplied from Çukurova Kimya. Glass fiber, were used as reinforcement and were purchased from Şişecam. Ceramic fiber, friction dust, graphite, petroleum coke, metal powder, barium sulfate were suppied from Zem Kimya. Sepiolite clay was supplied from Dolsan Minning, Eskisehir, Turkey.

2.2. Methods

2.2.1. Compositions of sepiolite clay containing composites

Composite samples containing different amounts of sepiolite clay were prepared. The formulations used to prepare the different samples are gathered in Table 1. In order to vary sepiolite clay and to maintain a constant volume at the same time, adjustments were made by decreasing filler content whith increasing sepiolite clay content.

2.2.2. Preparation and processing of sepiolite clay containing composites

The steps of the preparation and processing of sepiolite clay containing composites are schematically presented in Figure 1. Firstly, ceramic fiber and glass fiber were stirred at 1200 rpm into the high speed mixer for 1 min. Then, the other components were added and mixed for 5 extra minutes. Afterwards, the mixture was pressed with a hot press under a pressure of 50 bars. The temperature of the pre-heated mold was 90 °C and it was increased to 160 °C. Then, the samples were cured applying a pressure of 50 bars for 10 minutes. Lastly, the samples were post-cured at 180 °C for 1 hour.

Table 1. Formulations used to prepare the different composites.

Raw materials	**SP0**	**SP5**	**SP10**	**SP15**	**SP20**
Binders	15	15	15	15	15
Reinforcements	4.5	4.5	4.5	4.5	4.5
Lubricants	7.5	7.5	7.5	7.5	7.5
Friction modifiers	10	10	10	10	10
Metal powder	25	25	25	25	25
Fillers	38	33	28	23	18
Sepiolite clay	0	5	10	15	20

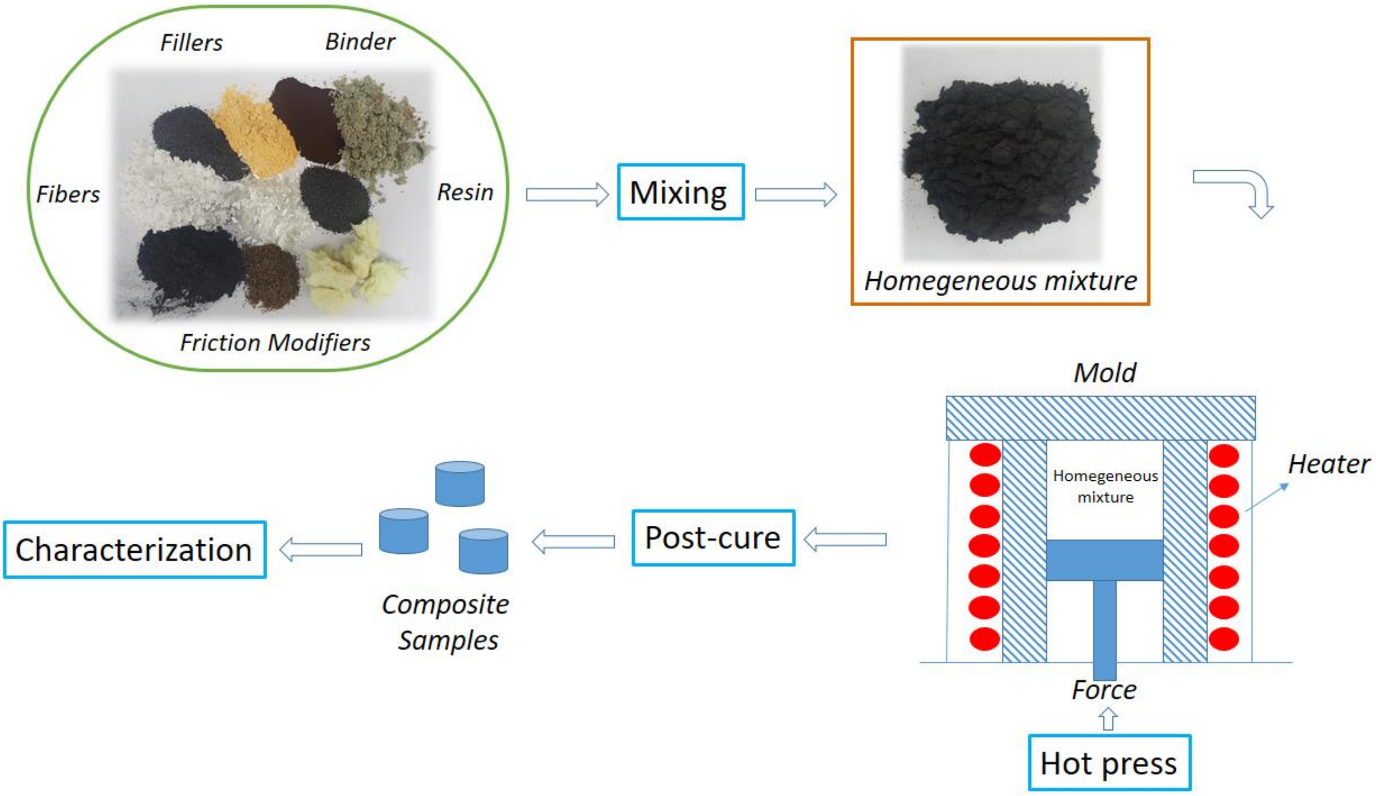

Figure 1. Schematic representation of the preparation of mixtures and processing of sepiolite clay containing composites.

Figure 2 presents a picture of the composite samples with different amounts of SP after post-curing. Due to the clear 'brownish' color of SP, the samples have a clearer color as SP content increases.

Figure 2. Photography of composite samples with different contents of SP after post-curing. The SP contents are indicated below each sample.

2.2.3. Characterization

The X-ray diffraction pattern of sepiolite clay was recorded with a Rigaku Miniflex 600 diffractometer (Cu tube, 1,54Å) with a scanning rate of 0.02°/s.

The morphology of sepiolite clay was observed by Scanning Electron Microscopy (SEM) using a Hitachi FlexSEM 1000 II microscope at a voltage of 5kV.

The density of the samples was determined from the mass and the volume of the samples.

The porosity was characterized based on the Archimedes principle. For this purpose, the procedure described in ASTM C373-88 standard was applied. According to this standard, the porosity P is expressed as follows:

$$P = \left[\frac{(M - D)}{M - S}\right] \times 100$$

where, P is the apparent porosity, D is dry mass, M is the saturated mass and S is the mass suspended in water.

After heating, the dry mass (D) of samples was measured using a precision balance. Then, in order to determine the other parameters, the samples were stored in distiled water for 3 days. After impregnation, the saturated mass (M) and mass suspended in water (S) were measured.

Shore D hardness of the samples was also measured using an analogue Shore D hardness tester at room temperature. Three replicates were used.

3. Results and Discussions

3.1. Properties of sepiolite clay

The morphology of sepiolite clay was characterized by SEM. The micrograph presented in Figure 3 clearly shows that it has a fibrous morphology. Sepiolite fibers of various length are observed. This observation is in correlation with the literature (Önal, Yılmaz, & Sarıkaya, 2008), (Yalçin & Bozkaya, 2004).

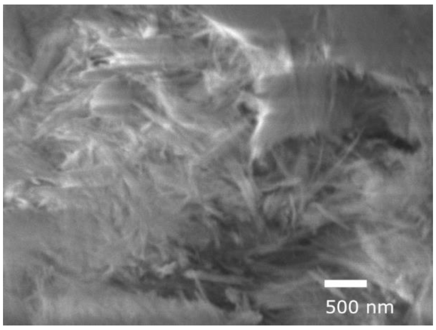

Figure 3. SEM micrograph of sepiolite clay.

The XRD pattern of sepiolite clay is also given in Figure 4. It can be observed that the two main characteristic peaks of sepiolite at 2θ ≈ 7° and 2θ ≈ 31° are present. This pattern is clearly in correlation with the literature (Moore & Reynolds, 1989), (Önal, Yılmaz, & Sarıkaya, 2008).

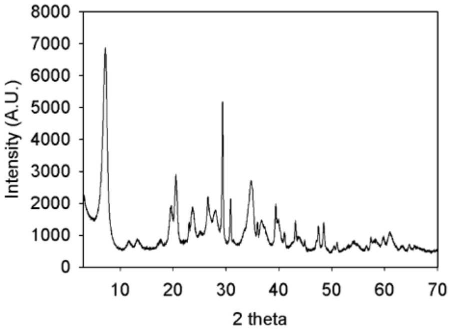

Figure 4. XRD pattern of sepiolite clay.

3.2. Effect of sepiolite clay content on the density of composite samples

As shown on Figure 4, the density of composite samples decreases almost linearly when sepiolite clay content increases. A maximum decrease of 5% is observed for sample containing 20% of sepiolite clay. This result can be expected because as mentioned in Section 2, the filler amount was decreased when the sepiolite clay content was increased in order to ensure that the total volume remains constant. As the density of fillers such as barium sulfate is higher than sepiolite clay, the density of the samples decreases with increasing SP content.

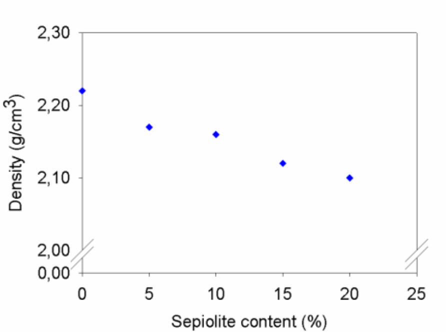

Figure 5. Evolution of the density of composite samples with varying sepiolite content.

3.3. Effect of sepiolite clay content on the porosity of composite samples

The porosity of the samples was characterized using the Archimed principle. The results are presented in Figure 5. According to this figure, the porosity of the samples decreases from 4.47% to 2.05% with increasing SP content until 15% of SP. Then, it rise to 2.81% for 20% of SP. This results are interesting and follow an opposite trend compared to the effect of potassium titanate fibers (Kim, Cho, Kim, & Jang, 2008).

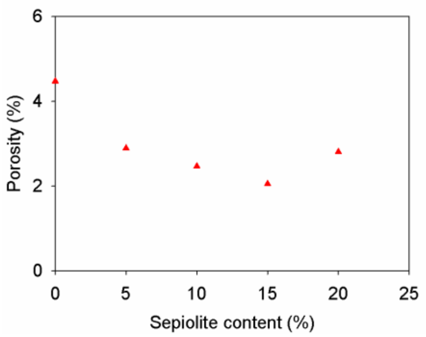

Figure 6. Evolution of the porosity of composite samples with varying sepiolite content.

3.4. Effet of sepiolite clay content on Shore D hardness of composite samples

The Shore D hardness results are gathered in Figure 6. The hardness increases with increasing SP content. This evolution may be attributed to the fibrous morphology of SP. Wannik et al. (Wannik, Ayob, Syahrullail, Masjuki, & Ahmad, 2012) observed the same tendency with samples containing boron friction modifier. Besides Yanar et al. (Yanar, Purcek, & Ayar, 2020) showed that the addition of steel fiber decreases the hardness.

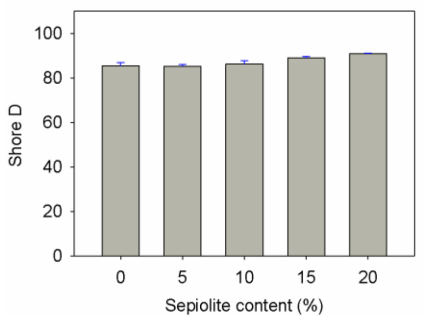

Figure 7. Evolution of the Shore D hardness of composite samples with varying sepiolite content.

4. Conclusion

In this study, effect of sepiolite clay mineral on processing and properties of novolac resin based organic composites for brake pad application was investigated. The main observation are that the hardness increases with increasing sepiolite clay amount. The highest Shore D hardness value was obtained with sample SP20. The density of composite samples decreased with increase in sepiolite clay content. The porosity decreases with increasing sepiolite content until 15% and rises again.

5. Acknowledgements

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