

Artículo de investigación

## Behavior of thermally treated kaolin filler in an asphalt concrete mixture

Comportamiento de un caolín tratado térmicamente como llenante mineral en una mezcla de concreto asfáltico

Comportamento de um caulim tratado termicamente como filer em uma mistura asfáltica quente

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- **Fecha de recepción:** 2019-03-28
- **Fecha concepto de evaluación:** 2019-10-31
- **Fecha de aprobación:** 2019-11-13  
<http://dx.doi.org/10.22335/rfct.v11i3.861>

**Para citar este artículo / To reference this article / Para citar este artigo:** Marín-García, B.F., Jiménez-Jiménez, O.J., & Rondón-Quintana, H.A. (2019). Behavior of thermally treated kaolin filler in an asphalt concrete mixture. *Revista Logos Ciencia & Tecnología*, 11(3), 10-17. <http://dx.doi.org/10.22335/rfct.v11i3.861>

### SUMMARY

In this study an industrial kaolin was subjected to high temperatures for one and two hours in order to eliminate plasticity and swelling properties. This heat-treated kaolin (HTK) was then evaluated for use as a substitute for natural mineral filler (passing No. 200 sieve; 6% by total mass of aggregate) in a Hot Mix Asphalt (HMA). The Marshall stability and flow test and Indirect Tensile Strength (ITS) test were performed to evaluate responses of HMA mixtures. X-ray diffractometry (XRD) and X-ray fluorescence (XRF) tests were carried out on kaolin (with and without thermal treatment). Effects of temperature and time of exposure on the penetration index (PI) and free swell index (FSI) of the HTK are also presented. Increased resistance under monotonic load and moisture damage was found when the natural mineral filler was completely replaced by the HTK. In contrast, when kaolin (K) that has not been heat-treated is used resistance and damage noticeably decrease especially in the presence of water.

**Keywords:** kaolin, filler, thermal treatment, hot mix asphalt



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## RESUMEN

En el presente estudio, un caolín de origen industrial fue sometido a altas temperaturas durante una y dos horas con el propósito de eliminar sus propiedades de plasticidad y expansión. Este caolín tratado térmicamente (KT) fue utilizado para evaluar su posible uso como sustituto de un llenante mineral (porcentaje pasa 200 en un ensayo de granulometría; 6% de la masa total del agregado) de origen natural en una mezcla de concreto asfáltico (HMA). Para evaluar la respuesta de las mezclas HMA fueron ejecutados ensayos de resistencia Marshall y tracción indirecta (ITS). Se realizaron pruebas de difracción de rayos X (DRX) y fluorescencia de rayos X (XRF) sobre el caolín (con y sin tratamiento térmico). También se presenta el efecto de la temperatura y el tiempo de exposición sobre los índices de penetración (PI) y expansión libre (FSI) de la KT. Como conclusión general se reporta un incremento en la resistencia bajo carga monotonía y al daño por humedad cuando espejo sustituye totalmente el llenante mineral natural por la KT. Contrario a lo anterior, si el caolín se utiliza sin tratar térmicamente (K), dichas resistencias disminuyen ostensiblemente, principalmente en presencia del agua.

**Palabras clave:** Caolín, llenante mineral, tratamiento térmico, concreto asfáltico

## SUMÁRIO

No presente estudo, um caulim de origem industrial foi submetido a altas temperaturas por 1 e 2 horas, a fim de eliminar suas propriedades de plasticidade e expansão. Este caulim tratado termicamente (KT) foi utilizado para avaliar sua possível utilização como substituto de um filler de origem natural em uma mistura asfáltica quente. Testes de resistência Marshall e tração indireta foram realizados para avaliar a resposta das misturas. Testes de difratometria de raios X (DRX) e fluorescência de raios X (XRF) foram realizados no caulim (com e sem tratamento térmico). O efeito da temperatura e tempo de exposição nos índices de penetração (PI) e expansão livre (FSI) do KT também é apresentado. Como conclusão geral, um aumento na resistência Marshall e dano por umidade é obtido quando o filler natural é completamente substituído pelo KT. Em contraste com o acima exposto, se o caulim é utilizado sem tratamento térmico (K), as referidas resistências diminuem ostensivamente, principalmente na presença de água.

**Palavras-chave:** Caulim, filler, tratamento térmico, mistura asfáltica quente

Clay soils, abundant in nature, are undesirable during road construction because they generally have low bearing capacities and shear strength, expand and to develop plastic behavior in the presence of water. They are usually discarded and sent to landfills, which negatively impacts the environment. On the other hand, the natural mineral filler used to produce asphalt mixtures is difficult to obtain because it is made up of particles whose sizes are less than 0.075 mm (passes sieve No. 200 in gradation test). In some cases, aggregates must be crushed to obtain filler, and this increases the cost of production and the consumption of energy.

Discarded clay soils can be used for many purposes including the manufacture of masonry, bricks (Abu-Zreig et al., 2001), ceramics (Yanti & Pratiwi, 2018) and geo-polymers (Elimbi et al., 2011). In this study is presented the possibility

of using heat-treated industrial kaolin as a filler in hot mix asphalt (HMA). Kaolin was subjected to high temperatures in order to eliminate its undesirable plastic and expansive behaviors that can negatively affect asphalt by decreasing resistance to moisture damage and stripping (Wasilewska et al., 2017). Industrial kaolin was chosen because this mineral is abundant in layers of clay soil but is more homogeneous than natural clays. This characteristic reduces the number of variables needed for evaluation.

A literature review found no studies of the use of thermally treated clays as fillers in asphalt mixtures. Some specially treated clays have been used as nano-materials for modifying the characteristics of asphalts (called nano-clays, Siddig et al., 2018). Several authors have found that incorporation of different proportions of nano-clays into asphalt has led to improvements in physico-mechanical

properties of asphalt mixtures such as increased resistance to rutting (Yu et al., 2007), resistance to cracking at low temperatures (Zare-Shahabadi et al., 2010) and resistance to fatigue (Ziari et al., 2014). Other authors have also reported satisfactory behavior of different types of treated clays in asphalt mixtures (Al Allam et al 2016, Mohd-Satar et al., 2018). These include clays previously stabilized with lime and then used in asphalt mixtures (Alizadeh and Modarres, 2019), synthetic coarse aggregate of calcined clay (SCACC) manufactured at high temperatures and used to replace the coarse fraction of asphalt mixtures (Lopes da Silva et al., 2015; De Souza Campelo et al., 2017, 2018; Losa et al., 2008), and waste bleaching clays from the food industry used as filler in asphalt mixtures (Sangiorgi et al. 2014, 2016). The last material did not have the typical properties of clay minerals. Bani-Baker et al. (2018) used a bentonite mixed into the mineral filler in proportions of 5, 10, 15, 20, 25 and 30% (by total weight of its portion of total aggregates). The bentonite, which is a highly plastic and expansive material, was not

heat-treated which may have resulted in undesirable long-term effects on the properties of the mixtures, but this was not evaluated.

## Materials and Methods

Asphalt concrete type HMA-19 (INVIAS, 2013) was chosen as the control material since in Colombia it is the type most commonly used for construction of surface or binder course asphalt layers of flexible pavements. For its manufacture, asphalt cement (AC) type AC-60-70 (penetration in 0.1 mm, ASTM D 5) and an aggregate that met the minimum quality requirements stipulated in INVIAS (2013) were used. The tests presented in Tables 1 and 2 were performed on both materials. The industrial kaolin (Figure 1) was yellowish white, its specific gravity was 2.68 (AASHTO T 84-00), its liquid limit (LL) was 39%, its plastic limit (PL) was 18% (plasticity index = 21%, ASTM D4318-

**Table 1.** Natural aggregate test results.

Test	Method	Recommended	Result
Specific gravity/fine aggregate absorption	AASHTO T 84-00	-	2.65/1.8%
Specific gravity/gross aggregate absorption	AASHTO T 85-91	-	2.54/1.5%
Sand equivalent test	AASHTO T 176-02	50% minimum	80%
Fractured particles (1 side)	ASTM D 5821-01	85% minimum	92%
Soundness of aggregate using magnesium sulphate	AASHTO T 104-99	18.0% maximum	9%
Plasticity Index	ASTM D4318-00	Not plastic	Not plastic
10% of fines (dry resistance)	DNER-ME 096-98	100 kN minimum	140 kN
Micro-Deval	AASHTO T327-05	20% maximum	17.6%
Abrasion in Los Angeles machine	AASHTO T 96-02	25% maximum	22.8%

Source: Authors.

**Table 2.** Results of tests performed on AC 60-70.

Test	Method	Unit	Recommended	Result
<b>Tests on original asphalt</b>				
Penetration (25°C, 100 g, 5 s)	ASTM D-5	0.1 mm	60-70	62.5
Penetration Index	NLT 181/88	-	-1.2/+0.6	-0.94
Dynamic viscosity (60°C)	ASTM D-4402	Poises	1500 minimum	1770
Softening point	ASTM D36-95	°C	48-54	49
Ductility (25°C, 5cm/min)	ASTM D-113	cm	100 minimum	>105
Flash and fire points	ASTM D-92	°C	230 minimum	289
<b>Tests on residue after Rolling Thin Film Oven Test</b>				
Mass loss	ASTM D-2872	%	0.8 maximum	0.63
Penetration (25°C, 100 g, 5 s)	ASTM D-5	%	50 minimum	77

Source: Authors.

00) and its free swell index (FSI) was 41.7% (IS: 2720, Part 40, 1977). Based on X-ray diffractometry (XRD), 77.1% by weight of minerals is natural Kaolinite, and the rest (22.9%) are Illite (20.6%) and Montmorillonite (2.3%). Based on X-ray fluorescence (XRF) tests, the chemical composition is shown below.

### Heat-treated Kaolin (HTK)

500 g Kaolin samples were placed in a laboratory muffle furnace at temperatures of 300, 400, 500 and 600 ° C for 1 and 2 hours. After heating, samples were left outdoors for one day to reach the laboratory ambient temperature of 20 ° C. These heat treated kaolin samples were named HTK. Their Atterberg limits (ASTM D4318-00) and FSI (IS:

2720, Part 40, 1977) were tested to determine the temperature and time of exposure at which the kaolin had completely lost all plastic and expansive behavior. This material was chosen as the replacement for the natural mineral filler. Three samples were tested for each run. The result presented is the average. Additionally, XRD and XRF tests were performed to evaluate mineralogical and chemical changes of the non-plastic and non-swelling HTK.

### Marshall and Indirect Tensile Strength Tests

The particle gradation (see Figure 2) recommended by INVIAS (2013) was used for manufacturing HMA-19. Marshall (AASHTO T- 245) and Indirect Tensile Strength - ITS (AASHTO T-283) tests were used to measure

**Table 3.** Results of tests performed on AC 60-70.

Test	Method	Unit	Recommended	Result
<b>Tests on original asphalt</b>				
Penetration (25°C, 100 g, 5 s)	ASTM D-5	0.1 mm	60-70	62.5
Penetration Index	NLT 181/88	-	-1.2/+0.6	-0.94
Dynamic viscosity (60°C)	ASTM D-4402	Poises	1500 minimum	1770
Softening point	ASTM D36-95	°C	48-54	49
Ductility (25°C, 5cm/min)	ASTM D-113	cm	100 minimum	>105
Flash and fire points	ASTM D-92	°C	230 minimum	289
<b>Tests on residue after Rolling Thin Film Oven Test</b>				
Mass loss	ASTM D-2872	%	0.8 maximum	0.63
Penetration (25°C, 100 g, 5 s)	ASTM D-5	%	50 minimum	77

Source: Authors.



**Figure 1.** Kaolin.  
Source: Authors

volumetric compositions of the mixtures (mainly air void content - Va) and resistance under monotonic load (using the Marshall quotient or stability/flow ratio - S/F). In the case of the Marshall test, five samples were manufactured for each asphalt percentages (in mass) of 4.5%, 5.0%, 5.5% and 6.0% in accordance with AASHTO T-245 guidelines. The temperatures of the samples for mixing and for compaction in the laboratory were 150°C and 140°C, respectively. These values were obtained based on the criteria established by the ASTM D6925 specification, where the viscosity required to obtain mix and compaction temperatures of HMA mixtures are 170 cP and 280 cP, respectively. These samples were manufactured with the aggregate and natural mineral filler (control HMAs). Other samples were prepared as described, but in one the natural mineral filler (passing No. 200 sieve; 6% by total mass of aggregate) was replaced by kaolin that had not been heat treated (designated K) while in the other one

it was replaced by HTK. These mixtures were designated HMA-19-K and HMA-19-KT, respectively.

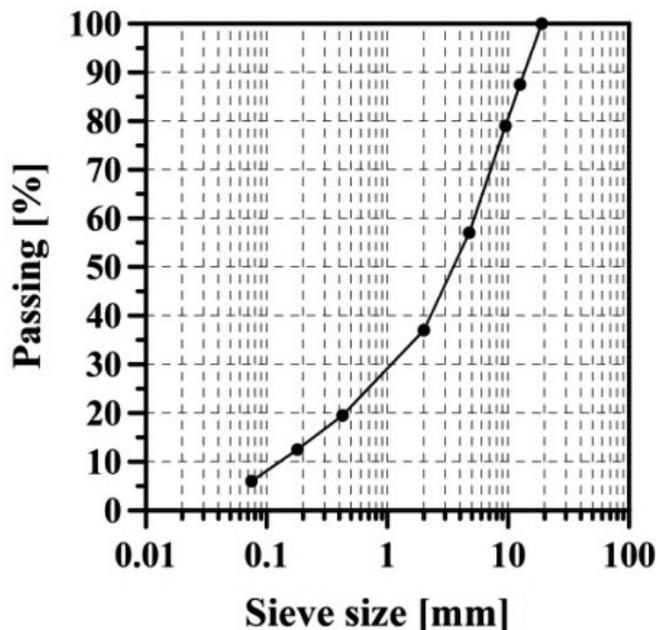


Figure 2. Particle size distribution of HMA-19.

Source: Authors.

Based on the Marshall test results, the optimum bitumen content (OBC) was also obtained. Using the OBC, new Marshall samples were manufactured in order to execute the ITS test. The samples for this test were manufactured as done for the Marshall test, but took into account that these samples had voids of  $7 \pm 1\%$  of their volume. HMA-19 (the control) and HMA-19-K and HMA-19-KT which used K and HTK as filler, respectively, were tested. Six samples for each mixture were tested at a temperature of  $25^\circ\text{C}$  in conditioned mixtures (ITS-W) and unconditioned mixtures (ITS-D) following AASHTO T-283 guideline specifications. In order to evaluate the resistance to moisture damage, the indirect tensile strength ratio TSR = ITS-D/ITS-W was calculated as a percentage.

## Results

### Heat-treated Kaolin (HTK)

In Table 3 are presented the results for tests performed on HTK. The Atterberg limits and the FSI decrease as temperature (T) and exposure time increase. When kaolin is calcined for two hours at a temperature of  $600^\circ\text{C}$ , it loses its plastic properties and its ability to expand. For this reason, the HTK obtained at this temperature and exposure time was chosen as the replacement for the

natural mineral filler (Figure 3). Figure 3 shows how the kaolin changed color when subjected to high temperatures. This change is due to physical-chemical changes that clay undergoes primarily as a product of water dissipation, decomposition of minerals (Geng & Sun, 2018) and changes in its micro-structure (Goodman et al., 2018). Chemical composition results for kaolin samples obtained from XRF tests are shown in Table 4. XRF results exhibit the increase in silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) in HTK compared to kaolin. This increase could positively affect microstructural strength. Silica ( $\text{SiO}_2$ ) is associated with high hardness and mechanical strength, and alumina ( $\text{Al}_2\text{O}_3$ ) presence implies good adhesion between aggregates and the asphalt binder (Modarres and Rahmanzadeh, 2014). Additionally, the  $\text{CaO}/\text{SiO}_2$  ratio is similar in all samples, that is, the affinity with the asphalt is also similar (Xie et al. 2012). Referring to the XRD analysis, the results for the temperature of  $600^\circ\text{C}$  revealed a considerable reduction of the kaolin, illite and montmorillonite minerals, keeping crystalline form quartz within HTK. This indicates an evolution towards a non-cohesive material, at least from a mineralogical point of view.

Table 3. HTK properties.

T ( $^\circ\text{C}$ )	LL (%)	LP (%)	IP (%)	FSI (%)
Time = 1 hour				
300	35	18	17	41.7
400	33	16	17	33.3
500	30	15	15	25.0
600	28	14	14	16.7
Time = 2 hours				
300	34	17	17	37.5
400	30	15	15	25.0
500	18	9	9	18.2
600	Non-plastic	Non-plastic	Non-plastic	0

Source: Authors.

Table 4. Chemical composition of natural filler, kaolin, and HTK ( $600^\circ\text{C}$  and 2 hours).

Material	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	CaO	$\text{K}_2\text{O}$	MgO	$\text{Na}_2\text{O}$	$\text{TiO}_2$	$\text{P}_2\text{O}_5$
Filler	61.90	10.82	5.58	1.55	1.23	3.02	2.22	1.00	0.10
Kaolin	61.16	18.92	3.71	0.12	1.70	1.67	0.91	0.53	0.95
HTK	67.82	22.32	2.67	1.36	1.10	0.89	0.88	0.24	0.67

Source: Authors.

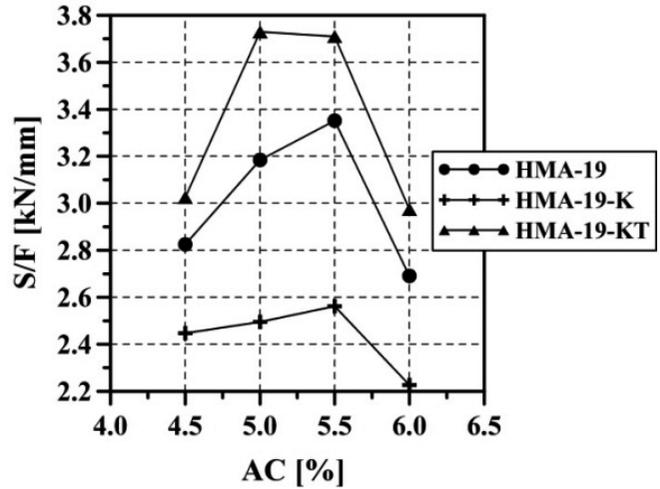


**Figure 3.** HTK at 600 ° C after two hours of exposure. Source: Authors.

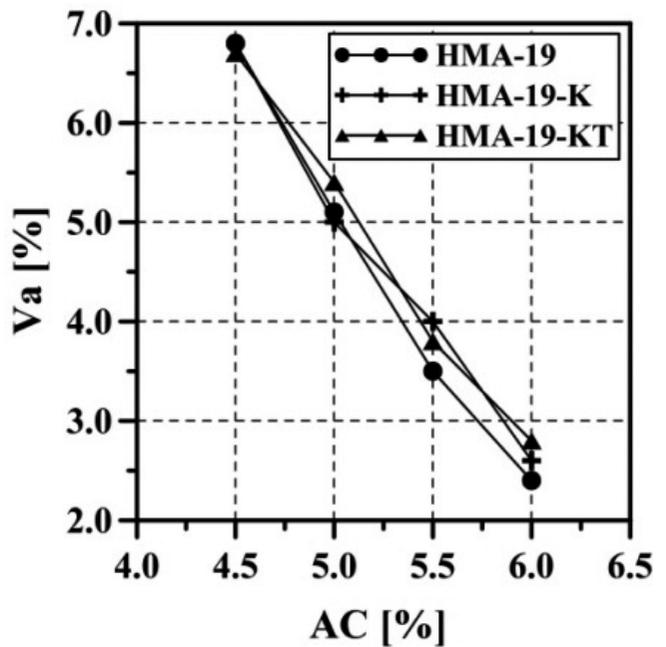
**Marshall and ITS Tests**

Figures 4 and 5 present Marshall test results. Resistance to permanent deformation of the asphalt mixes was measured by the Marshall Quotient (MQ), which is the ratio of stability to flow (S/F). Resistance increased between 7% and 17% when HTK replaced the natural mineral filler, but when K replaced the natural filler, resistance decreased significantly. In addition, it is important to note that the surfaces of the HMA-19-K samples became slimy and slippery on contact with hands when they were submerged in a 60 ° C water bath to perform the Marshall test. On the other hand, neither K nor HTK generate significant changes in the volume of air voids (Figure 5). The OBC, obtained on the basis of INVIAS guidelines (2013), was 5.3%. This asphalt content was used to manufacture the samples for the ITS test, the results of which are presented in Table 5. Mixtures that used kaolin without heat treatment as filler (HMA-19-K) were destroyed during the conditioning process to prepare them for the wet-condition ITS test in which samples were submerged at 60 ° C for 24 h. The ITS-W reported is zero because when kaolin without heat treatment is exposed to water inside the mixture, it reacts with the asphalt and its resistance to moisture damage decreases significantly. Nevertheless, dry unconditioned mixtures with untreated kaolin developed higher ITS-D resistance. This was because water does not enter the pores of the mixture in this condition so it cannot react with K. Increased resistance of ITS-D and ITS-W is also observed when HTK replaces the natural mineral filler. In other words, heat treatment

resulted in a filler material that helped increase the mixture’s resistance to moisture damage. This occurred primarily because heat treated kaolin K lost the undesirable properties that clays have in the presence of water.



**Figure 4.** S/F evolution. Source: Authors.



**Figure 5.** Air void content - Va. Source: Authors.

**Table 5.** ITS test results.

HMA type	Va [%]	ITS-D [kN]	ITS-W [kN]	TSR [%]
Control HMA-19	6.8	1047.1	875.7	83.6
HMA-19-K	7.1	1216.7	0.0	0.0
HMA-19-KT	7.2	1170.1	992.5	84.8

Source: Authors.

## ■ Conclusions

Experiments were used to evaluate how replacement of natural mineral filler with heat-treated kaolin (HTK) in an asphalt concrete mix affected the asphalt's mechanical strength under monotonic loads. Based on the results obtained, the following conclusions can be taken:

1. When the temperature and time of exposure increase, kaolin's plasticity and free expansion rate significantly decrease. The kaolin loses its properties of plasticity and expansiveness when subjected to a temperature of 600 ° C for 2 hours. Also, the chemical and mineralogical composition of kaolin changed for the better.
2. When all of the natural mineral filler is replaced with HTK, the mixture increases its resistance under monotonic loading (The S/F ratio and indirect tensile strength both increase). This indicates that resistance to permanent deformations probably increases in high temperature climates. Similarly, increased ITS and TSR indicate improved adherence between the asphalt and the aggregate and also indicate increased resistance to moisture damage.
3. The asphalt mixture's behavior was undesirable when the natural mineral filler was replaced with untreated industrial kaolin (K). The S/F ratio decreased significantly and when samples were heated at 60 ° C for 40 minutes in a water bath, they became slimy and slippery. In other words, in high temperature climates these mixtures could undergo significant permanent deformations. In addition, their resistance to sliding is likely to be low. In the case of the ITS-W in which samples were conditioned by being submerged in water at 60 ° C for 24 hours, the mixtures were completely destroyed. This indicates very low resistance to moisture damage.
4. The HTK had no significant effects on the void content of the mixture. Replacing the natural mineral filler with HTK could be a viable technique for manufacturing asphalt concrete mixtures. However, studies should be carried to analyze the use of natural clays. In addition, properties of mixtures under cyclic loading and the influence of the temperature on the chemical and mineral properties of the K and HTK analyzed must be further evaluated. In practice, some possible disadvantages in the methodology proposed would be energy consumption and possible emissions that could be generated during the kaolin's thermal treatment. Additional research on cost/benefit from a technical-social-environmental point of view should be carried out in this regard.

## ■ Reconocimientos

El artículo resultado de la investigación "Uso de arcillas tratadas térmicamente en mezclas asfálticas". Universidad Pedagógica y Tecnológica de Colombia y Universidad Distrital Francisco José de Caldas. 2018.

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