Use of Thermally Treated Bentonite as Filler in Hot Mix Asphalt

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Abstract: Clays and clay minerals are widely used as raw materials in different industrial processes due to their abundance. Generally, in road construction projects, clays are considered waste materials because they have some undesirable engineering properties (e.g., plasticity and swelling properties). For this reason, this material is generally discarded in landfills or dumps. However, taking advantage of its abundance, this material could be used as a substitute for natural aggregates of hot mix asphalt (HMA) production as long as their undesirable properties are eliminated. In this study, a bentonite was thermally treated (subjected to high temperatures) in order to be used as replacement for the total filler content of a natural aggregate in HMA. X-ray diffractometry (XRD), X-ray fluorescence (XRF), Atterberg limits, and free swelling index tests were carried out on bentonite (with and without thermal treatment). Marshall, indirect tensile strength (ITS) (under dry and wet conditions), resilient modulus, permanent deformation, resistance to fatigue, and Cantabro tests were carried out on HMA mixtures using thermally treated bentonite (BT) as filler. On all test results an ANOVA test was carried out. When BT replaced the total fraction of the natural filler, its resistance under monotonic load, stiffness under cyclic loading, and resistance to moisture damage increased remarkably. A similar resistance to fatigue and abrasion in the Cantabro test was observed. Based on the results obtained, BT could be considered a technically viable alternative as a substitute material for natural fillers in HMAs. **DOI: 10.1061/(ASCE)MT.1943-5533.0003127.** © *2020 American Society of Civil Engineers.*

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Introduction

Temperature Effects in Clays

Soil clays may exhibit viscous behavior, and their properties change with temperature (Abu-Zreig et al. 2001; Wang et al. 2016). Past studies evaluated the effect of high temperatures on the properties of swelling clays. Results showed that high temperatures changed their geotechnical properties (Laughlin 1959; Mitchell 1969; Towhata et al. 1993; Cho et al. 1999). When some clays are subjected to high temperatures, their physical, chemical, and thermal properties change (Joshi et al. 1994; Sun et al. 2016). According to Han et al. (2017) and Geng and Sun (2018), these changes are due mainly to the dissipation of water, the decomposition of minerals, and the combustion of organic matter, which also affect the clays' microstructural characteristics (Abu-Zreig et al. 2001; Sun et al. 2016; Goodman et al. 2018) and their pore structure (Živica and Palou 2016). Abuel-Naga et al. (2006) mentioned that several studies have demonstrated that high temperatures (above 100°C) affect the engineering properties of saturated fine-grained soils such as permeability, compressibility, stiffness, and shear strength. Likewise, several material characteristics, such as strength, cohesion, internal friction angle, and resistance to abrasion, are altered (Mitchell 1969). Additionally, high temperatures also help to decrease cation exchange (Joshi et al. 1994) and specific gravity (Tan et al. 2004; Zuzana et al. 2012), increase particle size (Yilmaz 2011), and, at firing temperatures, change the mineralogical composition (Sun et al. 2016; Panduro and Cabrejos 2010; Yanti and Pratiwi 2018).

Drits et al. (1995) concluded that, depending on the type of clay mineral, the temperatures at which the dehydration and dehydroxylation processes occur oscillate between 500°C and 800°C. A similar conclusion was drawn by Brindley (1975), but the established temperature range was 400°C–900°C. Zuzana et al. (2012) found that the dehydration of a natural bentonite was completed at 650°C. According to Abu-Zreig et al. (2001), Akbar-Firoozi et al. (2014), and Gadzama et al. (2017), high temperatures (above 100°C) have a significant effect in decreasing Atterberg limits and swelling pressure. A similar conclusion was arrived at by Shariatmadari and Saeidijam (2012), but testing a bentonite–sand mixture at temperatures between 25°C and 90°C. This decrease in Atterberg limits

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depends on the clay mineral type (Jefferson and Foss-Rogers 1998). Yilmaz (2011) found that kaolinite becomes nonplastic when kept for 24 h at 400°C and bentonite at 500°C. A similar conclusion was reported by Tan et al. (2004) for two types of clays that were kept for 24 h at 400°C.

Some Engineering Applications of Clays

Clays have been subjected to high temperatures to suit several engineering applications, such as geopolymer cements (Zibouche et al. 2009; Elimbi et al. 2011), bricks for dwellings (Joshi et al. 1994; Abu-Zreig et al. 2001), and ceramic products (Yanti and Pratiwi 2018), and to decontaminate soils from radioactive and toxic substances (Varlakov et al. 1997). In the case of bentonite, its greatest use is as nanoparticle or nanoclay for the modification of asphalts. Nanomaterials such as nanoclay are promising techniques for preventing moisture damage and improving the properties of bitumens and asphalt mixtures (Yusoff et al. 2014; Ashish et al. 2017; Akbari and Modarres 2018; Crucho et al. 2018; Omar et al. 2018; Siddig et al. 2018). According to Yu et al. (2007), asphalts modified with montmorillonite (MMT) clay and organically modified montmorillonite (OMMT) have higher rutting resistance and very good storage stability compared to conventional asphalts. An improvement in the dynamic properties (higher complex modulus, lower phase angle, and higher rutting parameter) and resistance to cracking of asphalt mixtures was reported by Zare-Shahabadi et al. (2010) when asphalt was modified with bentonite clay and organically modified bentonite (OMBT). Ziari et al. (2014) used 5% of bentonite (10%, 15%, 20%, 25%, and 30%) by weight of asphalt to modify it. Using the modified asphalt, they found that the fatigue life of hot mix asphalt (HMA) was longer and that the Marshall quotient (stability–flow ratio), indirect tensile strength, and resilient modulus was higher than those of the control mixture. Other types of mineral clays are also used for the same purpose. For example, Al-Allam et al. (2016) modified a binder with a soft clay (from Malaysia) using proportions of 2%, 4%, 6%, and 8% by weight. They obtained satisfactory results in the HMA for resilient modulus, moisture damage resistance, and dynamic creep tests, especially at 4%. In a similar study, Mohd-Satar et al. (2018) employed kaolin clay in the same proportions and obtained satisfactory results in Marshall tests, especially at 2%.

On the other hand, some studies have employed synthetic aggregates such as expanded clay or coarse aggregate of calcined clay (SCACC) in the production of HMAs. HMAs using SCACC have obtained good results in terms of physical and mechanical properties in comparison to conventional mixtures, mainly at higher temperature of aggregate calcination (da Silva et al. 2015; de Souza Campelo et al. 2018, 2019). Expanded clay could be used to increase friction performance and noise-spectrum reduction of asphalt surfaces (Losa et al. 2008). Other studies employed clays in asphalt mixtures, but the material had been treated in advance with lime (e.g., Alizadeh and Modarres 2019).

Problem Statement and Objective

Filler in asphalt mixtures is part of the aggregate gradation (particles smaller than 75 μm) and an important component that affects behavior (workability, moisture damage, fatigue and permanent deformation resistance, durability, aging, and long-term characteristics) of asphalt mastic and asphalt mixtures (Cheng et al. 2016). During road construction, a subgrade layer may be formed by clay soils (which are composed of particles smaller than 75 μm), and large amounts of these soils are usually disposed of as waste materials in landfills or dumps, negatively impacting the environment. Clays are generally considered waste materials due to some undesirable engineering properties, such as their susceptibility to plastic strains and swelling in the presence of water. This is mainly due to electrochemical forces that attract water firmly to the clay platelets, forming a water layer that is not able to flow under normal conditions (Ma and Hueckel 1992). In the case of asphalt mixtures, according to Chen et al. (2015) and Wasilewska et al. (2017), clay fines reduce the water resistance of bitumen–aggregate binding and resistance to moisture damage and cause stripping. These problems associated with clay minerals are due mainly to the presence of interlayer water molecules (which cannot be removed by air drying), which react chemically with asphalt, weakening the bond and even generating a spontaneous emulsification process (Scott 1978; Taylor and Khosla 1983; Little and Jones 2003; Chen et al. 2015). Other possible causes are associated with the cation exchange capacity and high specific surface area of the clays. Because of these facts, few studies have considered using clays as aggregates or filler in HMAs. Sangiorgi et al. (2014, 2016) used waste bleaching clays from the food industry as an alternative material to replace a common limestone mineral filler in HMA. The results obtained are interesting; however, this type of material does not present the typical properties of natural clays. Bani-Baker et al. (2018) used different bentonite contents (5%, 10%, 15%, 20%, 25%, and 30% by weight) to replace mineral filler. However, in this study, they only carried out Marshall, hydraulic conductivity, and indirect tensile strength (ITS) tests. Results showed that natural bentonite clay in an asphalt mixture increased the density and stability of mixtures containing 15% bentonite; however, there was a 23% loss when they were soaked in water for 24 h. The addition of bentonite also increased flow and ITS values, reaching maximum strength when the clay content was 10%.

This study, unlike previous ones, used a thermally treated bentonite (BT) to replace the entire natural filler (100% of total weight) in HMA. BT refers to bentonite that was subjected to high temperatures to eliminate its firmly adsorbed water, its properties of plasticity, and its swelling capacity, so that it could be used as filler. If these undesirable properties were eliminated, clay materials would have a substantial field of application. Not only could they be used as filler in HMA, but they could also serve as filler in stone mastic asphalts or mixtures using aggregates with predominantly fine particles. Additionally, they could serve as filler material for bases, subbases, and stabilized soils, for example. The authors found no references on the use of this material in the way it was used in the present study. In that sense, this could be presented as an alternative and innovative technique. An industrial bentonite was used because it is more homogeneous than natural clays. Additionally, bentonite is a clay mineral with high montmorillonite content that exhibits high water absorption and a high cation-exchange capacity (Uddin 2008).

Materials and Methods

The materials and testing methods employed are briefly described in this section. Materials include natural aggregates, an industrial bentonite, asphalt cement (AC), and hot mix asphalt (HMA). Laboratory procedures include conventional characterization tests, X-ray analysis, mix dosage, bentonite thermal treatment, and mechanical tests on the HMA samples (Marshall, ITS, resilient modulus, permanent deformation, fatigue resistance, and Cantabro tests).

To evaluate whether the natural filler substitution with BT produced statistically significant effects on HMA mix properties, an

Table 1. Natural aggregate test results

Test	Method	Recommended value	Result
Specific gravity/fine aggregate absorption	AASHTO T84	—	2.65/1.8%
Specific gravity/coarse aggregate absorption	AASHTO T85	—	2.54/1.5%
Specific gravity of soil solids by water pycnometer	AASHTO T100	—	2.66
Sand equivalent test	AASHTO T176	50% minimum	80%
Fractured particles (1 side)	ASTM D5821	85% minimum	92%
Soundness of aggregate using magnesium sulfate	AASHTO T104	18.0% maximum	9%
Plasticity index	ASTM D4318	Not plastic	Not plastic
10% fines (dry resistance)	DNER-ME 096	100 kN minimum	140 kN
Micro-Deval	AASHTO T327	20% maximum	17.6%
Abrasion in Los Angeles machine	AASHTO T96	25% maximum	22.8%

Table 2. Test results performed on AC 60-70

Test	Method	Unit	Recommended	Result
Tests on original asphalt				
Penetration (25°C, 100 g, 5 s)	ASTM D5/D5M	0.1 mm	60–70	62.5
Penetration index	NLT-352	—	−1.2/+0.6	−0.94
Viscosity (60°C)	ASTM D4402/D4402M	Poise	1,500 minimum	1,770
Softening point	ASTM D36	°C	48–54	49
Ductility (25°C, 5 cm/min)	ASTM D113	cm	100 minimum	>105
Flash and fire points	ASTM D92	°C	230 minimum	289
Tests on residue after rolling thin film oven test				
Mass loss	ASTM D2872	%	0.8 maximum	0.63
Penetration (25°C, 100 g, 5 s)	ASTM D5/D5M	%	50 minimum	77

ANOVA with an F-test was performed with a reliability level of 95%.

Materials

The natural granular aggregate and asphalt cement AC 60-70 [penetration test (ASTM 2013) at 0.1 mm] were characterized following the INVIAS (2013) standard. Lab test results are presented in Tables 1 and 2, respectively.

The tests performed on the AC 50-70 binder were penetration [ASTM D5 (ASTM 2013)], penetration index [NLT 181 (NLT 1988)], softening point [ASTM D36 (ASTM 2006)], viscosity [ASTM D4402 (ASTM 2015)], flash and fire points [ASTM D92 (ASTM 2001b)], ductility [ASTM D113 (ASTM 1999)], and the rolling thin-film oven test [ASTM D2872 (ASTM 2012)]. The tests performed on the natural aggregate were specific gravity and absorption [AASHTO T84 (AASHTO 2000); AASHTO T85 (AASHTO 1991)], specific gravity of soil solids by water pycnometer [AASHTO T100 (AASHTO 2003)], sand equivalent test [AASHTO T176 (AASHTO 2002a)], fractured particles [ASTM D5821 (ASTM 2001a)], soundness of aggregate using magnesium sulfate [AASHTO T104 (AASHTO 1999)], plasticity index [ASTM D4318 (ASTM 2000)], 10% fines [DNER-ME 096 (DNER 1998)], Micro-Deval [AASHTO T327 (AASHTO 2005)] and Los Angeles machine [AASHTO T96 (AASHTO 2002b)]. It is possible to observe that the values meet INVIAS (2013) requirements and specifications for the manufacture of HMAs in Colombia.

An industrial bentonite was employed with a specific gravity (AASHTO 2003) of 2.69 (Fig. 1). Based on X-ray diffractometry (XRD) and X-ray fluorescence (XRF) tests, 99.6% by weight of minerals is natural montmorillonite, and the rest (0.4%) is metahalloysite. Additionally, the chemical composition is shown in what follows. Bentonite has a plasticity index (*PI*) (ASTM 2000) of 343% and a free swell index (*FSI*) of 615.4%. Bentonite was classified as highly expansive and characterized as clay of high plasticity (CH) according to the Unified Soil Classification System (USCS).

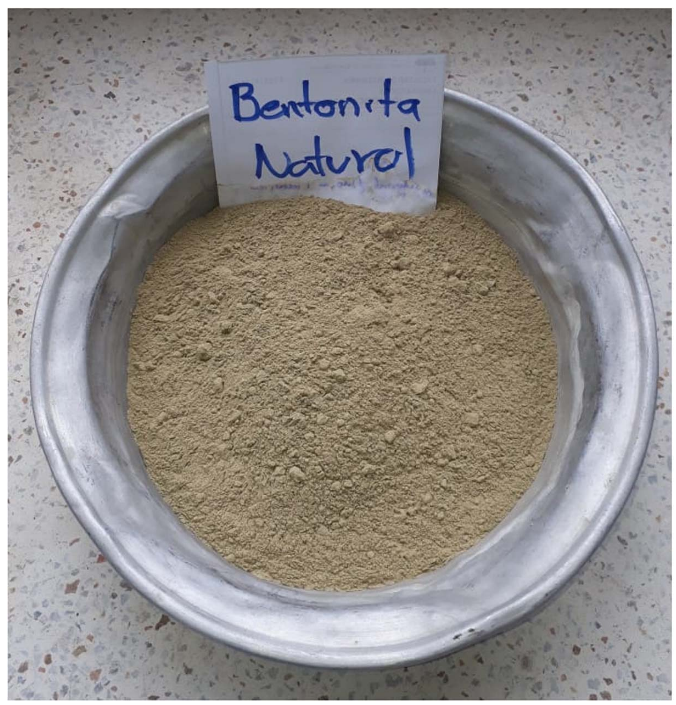

Fig. 1. Bentonite. (Image by authors.)

Asphalt Control Mix Design

The control HMA was designed based on a Marshall test (AASHTO 2015). Samples were compacted using 75 blows per face. The HMA granular size distribution is presented in Table 3 in accordance with INVIAS (2013) standards. The asphalt mixture was called HMA-19 (19 mm is the maximum nominal size of particles). To obtain the optimum asphalt content (OAC), five samples were manufactured and tested for each percentage of asphalt in

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Table 3. Particle size distribution of HMA-19

Sieve	Sieve (mm)	Percent passing (%)	Percent retained (%)
19.00 mm (3/4 in.)	19.00	100.0	0.0
12.50 mm (1/2 in.)	12.50	87.5	12.5
9.50 mm (3/8 in.)	9.50	79.0	8.5
4	4.75	57.0	22.0
10	2.00	37.0	20.0
40	0.43	19.5	17.5
80	0.18	12.5	7.0
200	0.075	6.0	6.5
Bottom	—	—	6.0

mass of 4.5%, 5.0%, 5.5%, and 6.0%. The mixing and compaction temperatures were 150°C (asphalt viscosity of 170 cP) and 140°C (asphalt viscosity of 280 cP), respectively. Additionally, the volumetric composition [air voids in volume (*Va*), voids in mineral aggregate (*VMA*), and voids filled with asphalt (*VFA*)] and resistance under monotonic load [stability (*S*), flow (*F*), and *S/F* ratio)] values were obtained.

Thermally Treated Bentonite (BT)

Bentonite samples of 500 g were burned in a laboratory muffle furnace at temperatures of 400°C, 500°C, 600°C, 700°C, and 800°C for 1 and 2 h. These samples were then left to cool at ambient conditions until a laboratory temperature of 20°C was reached. Within these samples (called BT) measurements were taken for Atterberg limits (liquid and plastic limits) to obtain a *PI* (ASTM 2000) and *FSI*. Three samples were tested in each test, and average values are reported as final results. These tests were carried out to determine the temperature and time required to eliminate plasticity and swelling properties in bentonite that would be used as filler in HMA-19. Additionally, XRD and XRF tests were performed to evaluate mineralogical and chemical changes in the nonplastic and nonswelling BT.

Marshall and Indirect Tensile Strength Tests

Marshall tests were performed on samples made with the OAC but replacing the total natural filler in mass (6% of the total aggregate, Table 3) with untreated bentonite (B) and with thermally treated bentonite (BT). HMA-19 results were called HMA-19-B and HMA-19-BT, respectively. Three samples for each mixture were manufactured to perform the Marshall tests. The manufacturing procedure and the mixing and compaction temperatures were the same as those used for the control HMA-19.

For the case of ITS tests, initially six Marshall-type samples were manufactured following the ASTM (2014) standard using the OAC and natural filler (control HMA-19). Three samples were tested with preconditioning (dry condition, ITS_D) and other three samples previously "conditioned" (vacuum saturated or wet, ITS_W). Moisture damage resistance was evaluated by means of the ratio $TSR = ITS_D/ITS_W$ (in percentage). Samples to be tested under wet conditions were placed in a saturation vacuum with a pressure of approximately 6.7 kPa for 5 min, and then specimens were submerged for 10 min in a water bath. Thereafter, the samples were submerged in a water bath at 60°C for 24 h. Prior to the ITS tests, the samples were submerged in water at 25°C for 2 h.

Additionally, another six samples (three tested in dry condition and three in wet condition) were manufactured and tested using the OAC and replacing the total natural filler with B and BT fillers (HMA-19-B and HMA-19-BT, respectively). These samples were tested following the same procedure described earlier, in the same way as control HMA-19.

Resilient Modulus, Permanent Deformation, and Fatigue Tests

Resilient modulus tests (BSI 2005c) were performed on control HMA-19 and HMA-19-BT mixtures under temperatures of 5°C, 20°C, and 40°C and three loading frequencies (2.5, 5.0, and 10.0 Hz). Tests were executed using a UTM-30 universal testing machine. Each resilient modulus test was carried out on three samples. Load amplitude was controlled to maintain a deformation level below 50 microstrain in accordance with BSI (2005c). This test was not performed on HMA-19-B due to its poor performance in the ITS tests.

Permanent deformation tests (BSI 2005b) under repeated square-wave load with a stress of 100 kPa, test temperature of 40°C, and 3,600 pulses (*N*) were performed on the Marshall HMA-19 and HMA-19-BT specimens used in the resilient modulus tests. The UTM-30 equipment was used to perform the tests. Prior to the tests, specimens were preconditioned in the thermostatic chamber at 40°C during 4 h. Then Marshall samples were preloaded under 10 kPa for 10 min. After preloading, a stress of 100 kPa was applied at $N = 3{,}600$ with 1 s, followed by a rest period of 1 s (frequency of 0.5 Hz).

Indirect tensile fatigue tests (ITFTs), according to (BSI 2005a) controlled stress procedure tests, were conducted on the HMA-19 and HMA-19-BT samples. Each fatigue test was performed using nine samples (three replicates for each of the three constant stress, σ, levels applied of 100, 250, and 400 kPa). Prior to testing, the specimens were placed in the thermostatic chamber and exposed to 20°C for 4 h. All fatigue tests were performed under a temperature of 20°C using the UTM-30 equipment, Marshall specimens [10.16 cm (4 in.) in diameter and 6.35 cm (2.5 in.) in thickness] and repetitive applications of compressive controlled load in a haversine waveform with a load time of 0.1 s and rest time of 0.4 s. Fatigue resistance was determined as the total number of load applications that caused the complete fracture of the specimen (N_f).

Cantabro Test

The Cantabro test is a useful tool for providing durability indices (generally including non-load-associated cracking and raveling or weathering) of dense graded asphalt mixes (Cox et al. 2017). Three Marshall-type samples of HMA-19 and HMA-19-BT mixtures were tested following NLT (1986). The same samples that were employed in the resilient modulus tests were used during the Cantabro tests (because resilient modulus is a nondestructive test). Each sample was tested at 20°C in a Los Angeles abrasion drum applying 500 revolutions without the loading of steel spheres. Mass loss in the samples was expressed as a percentage of the original sample mass and the final mass obtained after the test.

Results

Asphalt Control Mix Design

The results of Marshall tests on the HMA-19 control samples are shown in Table 4. The OAC was 5.3%, based on the requirements established by INVIAS (2013) for HMA-19 mixtures (minimum stability, *S*, of 9 kN; flow, *F*, between 2 and 4 mm; S/F ratio between 3 and 5 kN/mm; air void content, Va, between 4% and 6%; void spaces in mineral aggregate, VMA, minimum of 15%; and voids filled with asphalt, VFA, between 65% and 78%).

Table 4. Marshall test results of control HMA-19

AC (%)	S (kN)	F (mm)	S/F (kN/mm)	Va (%)	VMA (%)	VFA (%)
4.5	11.3	4.0	2.8	6.8	16.7	59.6
5.0	12.1	3.8	3.2	5.1	16.3	68.6
5.5	12.4	3.7	3.3	3.5	16.0	77.9
6.0	11.3	4.2	2.7	2.4	16.0	85.3

Table 5. Chemical composition of natural filler, bentonite, and BT (800°C and 2 h)

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	Na_2O	TiO_2	P_2O_5
Filler	61.90	10.82	5.58	1.55	1.23	3.02	2.22	1.00	0.10
Bentonite	48.08	19.08	6.37	1.26	0.76	3.04	2.62	0.88	0.09
BT	55.44	25.13	9.11	1.19	0.65	2.98	2.58	0.79	0.08

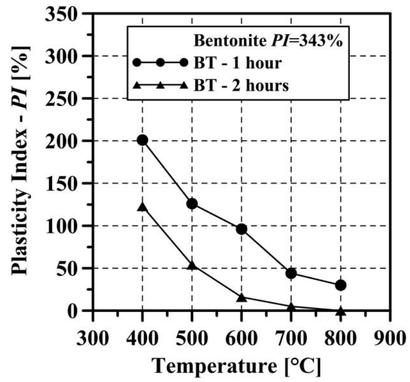

Fig. 2. PI evolution with temperature and time exposure.

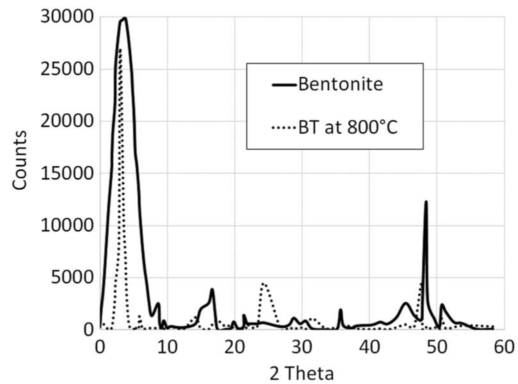

Fig. 4. XRD results.

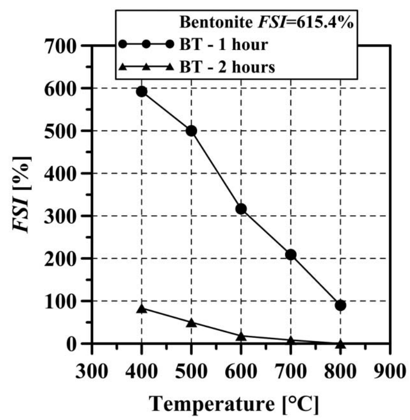

Fig. 3. FSI evolution with temperature and time exposure.

Fig. 5. Bentonite subjected for 2 h to (a) 1,000°C; (b) 800°C; (c) 600°C; and (d) industrial bentonite without thermal treatment. (Images by authors.)

Thermally Treated Bentonite

The specific gravity (AASHTO 2003) of the BT was 2.62. Its value is lower than the bentonite without thermal treatment of 2.69. This decrease is consistent with those reported by other researchers (e.g., Tan et al. 2004; Zuzana et al. 2012). Figs. 2 and 3 show the *PI* and *FSI*, respectively. It is possible to observe that both the *PI* and *FSI* decrease with an increase in temperature and with an increase in time of exposure. When bentonite is exposed to 800°C for 2 h, it becomes nonplastic and nonswelling ($PI = 0$ and $FSI = 0$). Therefore, the BT obtained at this temperature and time of exposure was chosen to replace the total natural filler of the HMA-19 in subsequent tests.

Chemical composition results for BT samples obtained from XRD and XRF tests are shown in Table 5. XRF results exhibit the increase in some oxides like silica and alumina in BT compared to bentonite without thermal treatment. This increase could positively affect microstructural strength. Silica (SiO_2) is associated with high hardness and mechanical strength, and alumina (Al_2O_3) presence implies good adhesion between aggregate and asphalt binder (Muniandy et al. 2013; Modarres and Rahmanzadeh 2014). Additionally, the CaO/SiO_2 ratio is similar in all samples (natural filler, B, and BT), that is, the alkalinity and affinity with the asphalt are also similar (Xie et al. 2012). Referring to the XRD analysis, shown in Fig. 4, the results at 800°C reveal a considerable reduction of the montmorillonite, retaining crystalline form quartz within the bentonite (BT). This indicates an evolution toward a noncohesive material, at least from a mineralogical point of view. These observed features are attributed to the physicochemical changes that clay undergoes mainly as a result of water dissipation, the decomposition of minerals (Geng and Sun 2018), and the change in its microstructure (Goodman et al. 2018). The effects of temperature on bentonite's physical appearance are also visually noticeable (bentonite color changes, Fig. 5).

Marshall and Indirect Tensile Strength Tests

Marshall and ITS test results are shown in Tables 6 and 7, respectively. When natural filler particles are replaced with bentonite without thermal treatment (HMA-19-B), the asphalt concrete

Table 6. Marshall test results

HMA type	S (kN)	F (mm)	S/F (kN/mm)	Va (%)	VMA (%)	VFA (%)
Control HMA-19	13.8	3.7	3.69	4.7	16.6	71.5
HMA-19-B	8.3	4.4	1.89	5.7	18.0	65.6
HMA-19-BT	17.2	3.9	4.43	5.3	17.3	68.1

Table 7. ITS test results

HMA type	Va (%)	ITS_D (kN)	ITS_W (kN)	TSR (%)
Control HMA-19	6.7	837.7	700.6	83.6
HMA-19-B	7.0	1,067.3	0.0	0.0
HMA-19-BT	7.2	1,115.2	951.3	85.3

undergoes a significant loss in resistance under monotonic loading in the Marshall test. Likewise, when samples are conditioned for ITS tests, the samples crumble and are easily destroyed ($ITS_W = 0$). This means that the HMA-19-B has no resistance to moisture damage. Additionally, the surface of the HMA-19-B samples acquired a smooth and slime consistency in contact with hands owing to the reactions that take place when samples are conditioned or submerged in water at 60°C (water is absorbed more easily into the sample and reacts with the bentonite). In spite of the foregoing considerations, if water does not enter the sample, HMA-19-B has a higher dry ITS strength compared to control HMA-19.

On the other hand, when BT is used as replacement for natural filler, the S/F ratio (Marshall quotient), ITS_D, ITS_W, and TSR ratio increase. The S/F ratio increased by 20% in relation to the control HMA-19 when BT was used as natural filler substitute. Additionally, this increase in the S/F ratio was statistically significant according to the ANOVA ($F = 35.5 > F_{0.05} = 7.71$). The HMA-19-BT with lighter BT presents a greater number of particles and therefore greater contacts and interlocking in the mineral skeleton. Additionally, the increase of silica oxides (SiO_2) also helps to enhance the Marshall quotient. For the case of ITS_D and ITS_W, increments were 33.1% and 35.8%, respectively. These increments were also statistically significant ($F = 176.4 > F_{0.05} = 7.71$ for ITS_D and $F = 155.7 > F_{0.05} = 7.71$ for ITS_W). Thus, thermal treatment also generated a material that helped to increase the moisture damage resistance of the HMA. The improvement observed is attributed mainly to the fact that BT lost those undesirable properties presented by the original clay in the presence of water. Additionally, when clays are submitted to high temperatures, their porosity and macropores increase (Živica and Palou 2016), which could also improve the adhesion properties of asphalt-aggregate.

The HMA-19-B and HMA-19-BT samples resulted in a slight increase in the void content (Va) in relation to the HMA-19 control mixture; however, the values obtained comply with the range, 4%–6%, established by INVIAS (2013), as also occurs with VMA values (15% minimum) and VFA (65%–75%). The increase in Va content in HMA-19-B is due to the greater specific gravity of bentonite compared to natural filler. In contrast to the natural bentonite mixture, HMA-19-BT should have lower Va compared to HMA-19 (due to the lower specific gravity). However, the loss of structural water in bentonite may also cause an increase in pores and fractures (increasing the number of particles), since some minerals are decomposed and evaporated (e.g., the dehydroxylation of clay minerals), according to Sun et al. (2016). Additionally, the removal of adsorbed water around particles leads to particle growth to larger sizes (Yilmaz 2011).

Resilient Modulus

The results of the resilient modulus tests are shown in Fig. 6. They are consistent with those reported from the Marshall tests. The HMA mixture with BT tends to show higher stiffness under cyclic loading at any temperature when compared to the HMA-19 control sample. The increment in the resilient modulus fell in the ranges 9%–10%, 21%–31%, and 21%–28% at 5°C, 20°C, and 40°C, respectively. ANOVA showed that BT had a significant effect on the resilient modulus for each temperature evaluated ($F = 31.9$ to $69.7 > F_{0.05} = 7.71$). In a similar way to the Marshall test, HMA-19-BT presented a greater number of particles compared to the HMA-19 control, generating a greater level of contact between particles. The resilient modulus may have increased for that reason. Additionally, the increase in particles with greater hardness (SiO_2) also helps to elevate the resilient modulus.

Permanent deformation results are presented in Fig. 7. The results show that the greater resistance to permanent deformation under cyclic loading is achieved by the HMA mixture with BT. This result is consistent with those obtained in Fig. 6 for the case of the resilient modulus. ANOVA on the permanent deformation test was calculated at $N = 3{,}600$ (final load cycle). This showed that BT has a significant effect on the measured axial displacement ($F = 38.22 > F_{0.05} = 5.14$).

Fig. 8 shows that HMA-19-BT develops a slight increase to fatigue resistance in comparison with the HMA-19 control mixture.

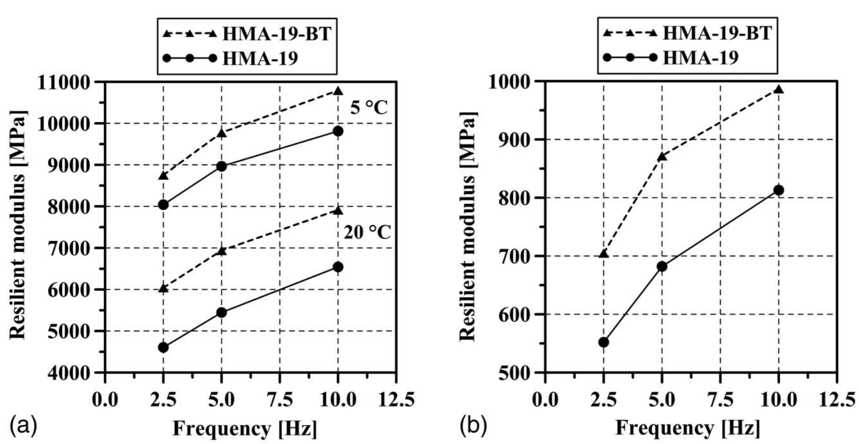

Fig. 6. Resilient modulus test results at (a) 5°C and 20°C; and (b) 40°C.

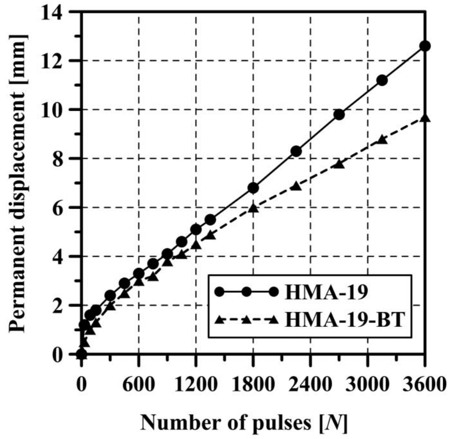

Fig. 7. Permanent deformation test results.

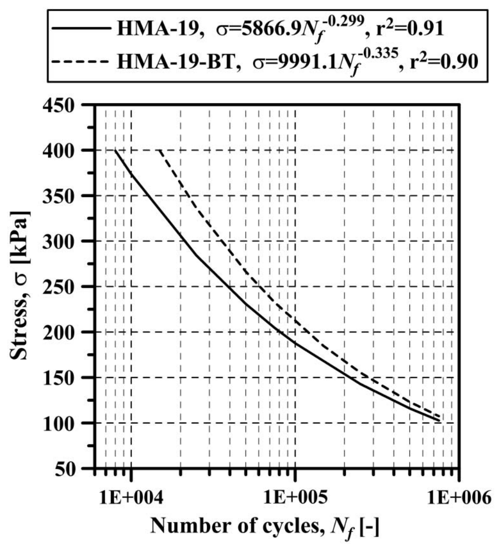

Fig. 8. Fatigue test results.

Table 8. Cantabro test results

Mixture	Va (%)	Mass loss (%), 500 revolutions
Control HMA-19	4.6	12.2
HMA-19-BT	5.1	11.8

However, the ANOVA calculated for stress levels of 100, 250, and 400 kPa shows that BT does not produce a significant effect on fatigue resistance ($F = 1 < F_{0.05} = 7.71$, $F = 5 < F_{0.05} = 7.71$, and $F = 3.8 < F_{0.05} = 7.71$ for 100, 250, and 400 kPa, respectively). This slight increase in fatigue resistance can be related to the mixture's response to this type of loading (controlled stress). When mixture stiffness increases with this type of loading, usually the same happens with its useful life load fatigue resistance. In contrast, when controlled deformation loading is imposed, usually greater fatigue life occurs when the mixture is less stiff (Di Benedetto et al. 2004; Muniz de Farias et al. 2019).

Cantabro Test

The results of the Cantabro tests are shown in Table 8. In comparison to the HMA-19 control, HMA with BT is a material with similar Cantabro mass loss and resistance to abrasion. Additionally, they are not statistically significant ($F = 5.04 < F_{0.05} = 7.71$).

Conclusions

In this research, an industrial bentonite was thermally treated (BT) for use as replacement for the total natural filler in a dense HMA. Based on the results obtained, the following conclusions can be drawn specifically for the BT analyzed:

- The *PI* and *FSI* of bentonite decrease with temperature increment and exposure time. Bentonite becomes nonplastic and nonswelling when it is kept for 2 h at a temperature of 800°C.
- Untreated industrial bentonite cannot be used as filler, since it significantly reduces the resistance of HMA to moisture damage and also reduces the mixture's Marshall ratio (S/F).
- The replacement of natural filler with BT in HMA slightly increased the air void content and increased mechanical resistance under monotonic loading (S/F ratio, ITS_D, and ITS_W) and moisture damage resistance (ITS_W and TSR). The volumetric composition of HMA with BT as filler meets the volumetric requirements for *Va*, *VMA*, and *VFA* established by the reference standard of INVIAS (2013).
- The resilient modulus is directly correlated with stiffness under cyclic loading and the permanent deformation resistance of HMA mixtures. A significant increase in stiffness under cyclic loading at 5°C, 20°C, and 40°C was obtained when BT replacd natural filler in the HMA. Additionally, an increase in resistance to rutting was obtained.
- Similar fatigue and Cantabro abrasion resistance was obtained when BT was used as filler compared to the control HMA.
- In general terms, the thermal treatment performed on the industrial bentonite was beneficial because it eliminated the adsorbed and interlayer water that adhered to the bentonite, thereby removing its undesirable properties (plasticity and swelling properties). In addition, the chemical and mineralogical composition of bentonite changed for the better. Finally, it is concluded that this treated material could be used to replace the filler fraction of natural aggregates in HMAs so as to improve the mechanical properties measured in the foregoing study. Additionally, it is highlighted that to obtain these results, it was not necessary to increase the OAC. In practice, some possible disadvantages in the proposed methodology would be energy consumption and possible emissions that might be generated during the thermal treatment of bentonite. Additional research on costs and benefits from a technical-social-environmental point of view should be carried out in this regard.

Data Availability Statement

Some or all data, models, or code generated or used during the study are available from the corresponding author by request.

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