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# Performance of nanoceramic powder on the chemical and physical properties of bitumen

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#### HIGHLIGHTS

• Addition of NCP into virgin bitumen can increase viscosity.

• Mixing of NCP into original bitumen can produce a polyaromatic structure system.

• Nano ceramic powder as bitumen modification has better rutting resistance.

#### ARTICLE INFO

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#### ABSTRACT

Approximately 30% of industrially produced ceramic materials worldwide are dumped as waste in landfills. Thus, these materials are a major environmental concern. Reusing or recycling these ceramic wastes for constructive purposes remains challenging. In this work, we evaluated for the first time the modified chemical and physical properties of bitumen incorporated with inorganic nanoceramic powder (NCP) produced from the ceramic wastes. Chemical and morphological properties of bitumen with NCP were determined. Bitumen specimens were characterized using transmission electron microscopy, X-ray fluorescence spectroscopy, X-ray diffraction, Fourier transform infrared spectroscopy, and atomic force microscopy (AFM). Physical and rheological properties of the specimens were also examined by penetration, softening point, and dynamic shear rheometer tests. Morphological analysis revealed the formation of an interaction between the Si—O—Si stretching vibration and hydrocarbon molecules. This interaction resulted from the incorporation of NCP into the unmodified bitumen matrix. The interaction increased the viscosity and reassembled the asphaltene particles. Thus, a new asphaltene sheet was generated, and the aromatic bonds (C=C) were strengthened. AFM results reaffirmed the structural improvement of bitumen. Furthermore, the elasticity of bitumen and viscoelasticity of its matrix increased. These improvements can be attributed to the formation of the polyaromatic structure.

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#### 1. Introduction

The general properties of conventional bitumen require enhanced endurance. Heavy traffic load and adverse climatic conditions, are responsible for the premature degradation (hightemperature-related rutting) of bitumen coatings [1,2]. An ideal bitumen binder should generally possess high relative stiffness at extreme service temperatures (especially during summer) to reduce the rutting and shoving and lead to strong adhesion between asphalt and aggregate in the presence of moisture to

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http://dx.doi.org/10.1016/j.conbuildmat.2017.09.014 0950-0618/© 2017 Elsevier Ltd. All rights reserved. reduce stripping [3]. Minimizing these concerns is remarkably economical for recovery, and reconstruction of defects would be expensive. This failure-related limitation can be overcome by modifying the properties of bitumen binder. Various methods, such as the use of diverse polymer types, to modify bitumen binders have been developed. Nanomaterials have been widely used as additives to improve the performance of bitumen binder and mixture.

Nanocomposites have extensively applied to enhance the properties of asphalt mixture [4–7]. Various types of nanomaterial are available, and each type offers different opportunities for modifying and improving asphalt mixtures. Nanomaterials used in asphalt applications include nanotubes [8], nanofibers [9], nanohydrated lime [10], nanosilica [11], polymerized powders, nanosized plastic







powders, and nanoclay [12]. Nanoclay with chemical composition of 67.70% SiO<sub>2</sub>, 23.3% Al<sub>2</sub>O<sub>3</sub>, and 3.78% Fe<sub>2</sub>O<sub>3</sub> significantly improves the engineering properties of asphalt binders and mixtures [13]. Furthermore, this material exhibits several benefits and prospects for modifying the behavior of base bitumen, especially for flexible pavement applications. This material has also been used as a secondary modifier to develop the performance properties of styrene-butadienestyrene (SBS)-modified binder [14].

Water sensitivity and tensile strength were studied with nanoclay and carbon microfiber modification. Nanomaterials can remarkably improve the mechanical strength of the asphalt mixtures, and modification with nanoclay (1.5%) results in high tensile strength and moisture susceptibility performance [15]. However, alternative modifier materials have attracted attention because of the continuous increase in the cost of bitumen and overall cost of modified bitumen with a typical additive. Modified bitumen with commercial additives usually increases the cost of the original bitumen (OB) between 60% and 150% [16,17]. Thus, using recycled materials as feasible bitumen modifiers can be an economical alternative to typical modifiers. In addition, recycled materials as bitumen modifiers are advantageous over traditional one. Many cases result in open pits, thereby causing negative impacts on the environment [18,19].

Ceramic materials are extensively used worldwide such that large quantities of wastes are inevitably produced by brick and tile manufacturing industries and construction sectors. A part of these wastes is dumped in landfills [20]. Notably, ceramic wastes are durable, hard, and highly resistant to biological, chemical, and physical degradations. Thus these materials cannot be recycled via existing processes [21]. In addition, approximately 30% of products in the ceramic industry are regarded as waste [22]. To date, the utilization of these wastes as modifier for bitumen binder is rarely studied. Moreover, few studies used different industrial waste materials as fillers in hot mix asphalt [23,24]. Muniandy et al. [25] reported the improvement of stiffness and rutting resistance by replacing 10% of conventional limestone filler with ceramic waste filler.

Given the notable benefits of nanoceramic powder (NCP) in improving material properties, the overall behavior of bitumen was modified by incorporating waste ceramics obtained from floor and wall tiles. NCP with varying concentrations (2%, 4%, and 6%) were used to modify the OB. The chemical composition of the NCP was analyzed by X-ray fluorescence (XRF) spectroscopy. The morphology of NCPs (i.e., nanoparticle size and shape) was determined by transmission electron microscopy (TEM). X-ray diffraction (XRD) was used for structural analysis. The broadening XRD peak revealed the effect of grinding on the nanocrystallite (grain) size and lattice parameters of NCP. The XRD pattern of the OB (without NCP inclusion) and modified bitumen (with varied contents of NCP inclusion) was further used to investigate the effect of NCP on the bitumen matrix. Furthermore, penetration, softening point, and dynamic shear rheometer (DSR) tests were conducted to examine the effects of NCP on the binder performance of asphalt. The variation in the chemical bonds was studied using Fourier transform infrared (FTIR) spectroscopy. Atomic force microscopy (AFM) was performed to determine surface morphology. The influence of adhesion/cohesion on the formation of microstructure and the appearance and surface coverage of the different nanocrystalline phases of the OB and modified bitumen were analyzed using AFM data. Inclusion of a small amount of NCP in the bitumen compared with the total volume of the mix remarkably improved the overall properties of the material. The results confirmed that safe use of NCP in asphalt concrete may be considerably beneficial for durable construction and sustainable development. The designed mixture can fulfill the requirement of environmental impact tests by the toxicity characteristic leaching procedure (TCLP) of EPA.

#### 2. Experimental

#### 2.1. Materials

Bitumen with 60/70 penetration grade was used as control asphalt binder. The binder source, which satisfied all the requirements of the Jabatan Kerja Raya (Malaysian standard), was obtained from Wira Bakti Solution Company (Malaysia). NCPs were used to modify the bitumen binder. These NCPs (as waste ceramics) were collected from a factory in Malaysia that produces floor and wall tiles for construction.

#### 2.2. Sample preparation

#### 2.2.1. Production of NCP

The collected waste ceramic powders from the factory was first manually crushed into small pieces and ground into a specific powder size using jaw crushing and LAAV machine. Subsequently, the powder was sieved to 75  $\mu$ m. The powder was heated in an oven at 130 °C for 30 min and ground for 5, 10, and 15 h using a bowl mill machine to determine the optimum grinding time.

#### 2.2.2. Bitumen modification

A stainless steel container ( $\approx$ 700 mL) was filled with 400 g of 60/70 grade base bitumen and placed in a thermoelectric heater. When the bitumen temperature reached 145 °C (i.e., upon achieving the required viscosity), a high-shear mixer (Silverson L5M) was dipped into the container and set to 4000 rpm. The NCPs were also heated to the same temperature to ensure the dryness of the powder. First, NCPs with changing concentrations (2%, 4%, and 6%) were mixed according to the weight of the binder and gradually added to avoid agglomeration, during which the temperature was maintained within the range of 145 ± 5 °C. Mixing was continued for approximately 90 min to uniformly disperse the NCPs into the bitumen matrix.

#### 2.3. Characterizations of samples

#### 2.3.1. XRF

The chemical composition of the NCPs was determined by XRF analysis. NCP was maintained in a desiccator for 8 h to completely remove moisture. Consequently, 3 g of the specimen was placed in a sample holder and analyzed by EDXRF under helium gas flow.

#### 2.3.2. TEM

The morphology of the NCPs (shape and size of nanoparticles) was examined using a transmission electron microscope (Hitachi HT7700) with high image resolution ( $\approx$ 1 nm) and magnification.

#### 2.3.3. XRD

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The broadening of the XRD peak, which is called the full width at half maximum (FWHM), was analyzed to determine the influence of grinding on the nanocrystallite grain size and lattice structure of the NCPs. This method is useful because of its dependability, convenience, and less time consumption [26]. The FWHM is related to the nanocrystallite sizes (*D*) through the Debye–Scherer equation as follows [27]:

$$\mathbf{D} = \kappa \lambda \beta \cos \theta \tag{1}$$

where  $\lambda$  is the wavelength of the X-ray radiation, k is a constant taken as 0.89,  $\beta$  is the FWHM of line broadening, and  $\theta$  is the angle of diffraction. The microstrain ( $\varepsilon$ ) involved in the modified bitumen grain was estimated using the following equation:

$$=\beta 4\tan\theta. \tag{2}$$

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