



## Case study

# The performance of Agbabu natural bitumen modified with polyphosphoric acid through fundamental and Fourier transform infrared spectroscopic investigations



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## ABSTRACT

Nigeria is greatly endowed with large deposit of natural bitumen at Agbabu, Ondo State. The country was reported to have a proven reserve of about 42.47 billion tonnes of bitumen, a quantity which is estimated to be second largest in the world but yet to be explored for economic purposes. However, literature has shown paucity of research work on its modification. This study is therefore carried out to investigate the suitability of polyphosphoric acid (PPA) as a modifier in Agbabu natural bitumen (ANB). The performance of unmodified and PPA modified ANB has been comparatively studied with specific emphasis on Fourier Transform Infrared investigation and fundamental physical tests like penetration, softening point, fire and flash point and specific gravity. Kinematic viscosity (flow) test was also investigated. Penetration indexes of ANB samples were calculated from the penetration values for softening points.

The comparative study of FTIR spectra of both base ANB and PPA modified ANB samples showed appearance of some few new peaks in the infrared spectra of PPA modified ANB samples confirming that the structure of the neat ANB had been modified. On the basis of our experimental results, incorporation of PPA up to 6% into ANB structure greatly enhances the fundamental physical and flow properties of ANB. This showed that PPA has the potential of improving the service life of ANB.

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## 1. Introduction

One of the major applications of bitumen is in road pavement construction [1]. However, experience has shown that application of bitumen in its natural form in pavement did not meet the required service life expectancy. The reason is attributed to increasing traffic volume on the highway and degradation of the binder (bitumen) used for the pavement. In order to meet the rising requirements for durability of the road surfaces, alternative methods of improving bitumen have been developed. One of the methods is the incorporation of modifiers which include organic and inorganic materials such as styrene butadiene (SB), styrene-butadiene-styrene (SBS), ethylene terpolymer, Ethylene-vinyl-acetate (EVA), Poly-ethylene (PE), Styrene-isoprene-styrene (SIS) and polyphosphoric acid [2–5]. The essence of this is to improve its rheological

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properties such as elasticity, viscosity as well as fundamental properties such as penetration value, softening point and flash and fire points prior to its application.

Polyphosphoric acid (PPA) is an acid produced by heating phosphorus pentoxide ( $P_2O_5$ ) with phosphoric acid ( $H_3PO_4$ ) [5]. It is a viscous liquid at 25 °C, highly soluble in organics and non-oxidising molecules, [5]. PPA is produced mainly in China and in the USA in various grades containing different amount of phosphoric acid ( $H_3PO_4$ ) or phosphorus pentoxide ( $P_2O_5$ ). To produce PPA modified bitumen, concentration of 105, 110 and 114%  $H_3PO_4$  are used (75.9, 79.8 and 82.6%  $P_2O_5$  respectively) [6]. Qunshan and Hongyuan [7] also reported a typical grade of PPA for bitumen containing 115% orthophosphoric acid. The grade contain no water.

The discovery of PPA as a suitable modifier to improve fundamental and high temperature rheological properties of bitumen without adversely affecting its low temperature rheological properties dated back to early 1970s [8]. Since then, there has been avalanche of publications on the use of PPA for investigation on bitumen. Some of these include: low temperature rheology of polyphosphoric acid added to bitumen by Baldino et al. [9]; rheological effect on bitumen of polyphosphoric acid addition by Baldino et al. [10] and performance of VG30 paving grade bitumen modified with PPA at medium and high temperature regimes by Ramayya et al. [11]. Their findings revealed that the stiffness of the bitumen increased as the percentage of PPA increased up to a value that can be considered critical and destabilizing, PPA addition improved mechanical performances of bitumen at high temperature and PPA addition enhanced performance grade (PG) of VG30 binder respectively. Masson and Collins (12) reported that the reaction of PPA with bitumen occurs at the site of heteroatom containing compounds. Sulphur which is predominantly the heteroatom in bitumen had been reported to be inert either in aliphatic or aromatic sulphide group when heated with PPA at 150 °C for 1 h. However, the reactivity of PPA with bitumen is a function of nitrogen and oxygen content [13] and the reaction mechanisms include: acidolysis of alkyl/ aromatics and nucleophilic displacement; alkylation of aromatics with sulphides and alcohols; and cyclisation of carboxylic acids amongst others [4].

Nigeria is ranked as one of the first five countries in the world endowed with largest deposit of natural bitumen [14], however, research activities on the vast deposit of Agbabu natural bitumen in Nigeria have largely been concentrated on the physico-chemical and engineering characterization [15]. Our aim in this study was therefore to investigate the potential of PPA in improving the service life of raw ANB.

## 2. Methodology

### 2.1. Materials

#### 2.1.1. Bitumen

The bitumen used in this study was natural bitumen sample collected from one of the observatory wells sunk by the Nigerian Bitumen Corporation (NBC -7) located opposite Saint Stephen's Primary School, Agbabu, Ondo State, Nigeria. Agbabu is located on the so called bitumen belt of south-western Nigeria. The belt lies within latitude  $006^{\circ}38'N$ – $006^{\circ}40'N$  and longitude  $004^{\circ}34'E$ – $004^{\circ}37'E$ , falls within the eastern Dahomey Basin and spans across Edo, Ondo and Ogun States [16,17].

#### 2.1.2. Modifier

The modifier used in this study was Polyphosphoric acid (PPA) with concentration of 105% phosphoric acid (75.9%  $P_2O_5$ ). The specific gravity is 2.05.

### 2.2. Preparation of PPA modified ANB

The raw natural bitumen sample collected from Agbabu was dehydrated and then purified as described in our previous study [18]. The PPA modified samples were prepared using a high shear mixer. A certain quantity of ANB (400 g) was heated to fluid state in an iron container. When the temperature of ANB was in range of 150–155 °C, the PPA was gradually added. The temperature of the mixture was maintained at 150–155 °C with the speed of the mixer also maintained at 1200 rpm for 1hr to obtain a homogenous mixture. The proportion by weight of PPA added to the base ANB varied from 2 to 6%.

### 2.3. Rheological properties of modified and unmodified ANB

The following rheological properties of the PPA modified ANB were determined using appropriate standard procedures. They are: Softening point [19], Penetration value [20], Kinematic viscosity [21], Flash and fire point tests of ANB [22].

#### 2.3.1. Penetration index of PPA modified and unmodified ANB

The response of modified bitumen samples to the effect of temperature changes was calculated in terms of penetration index (PI) using the results obtained from penetration and softening point tests.

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