BITUMEN-AGGREGATE ADHESION: A PREDICTIVE STUDY BASED ON ZETA POTENTIAL ANALYSIS USING THE STREAMING POTENTIAL TECHNIQUE

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ABSTRACT

Bitumen and aggregates are the main constituents of asphalt; their physical and chemical properties have a direct influence on the performance of the mixture. Adhesion between mineral aggregates and bitumen is an important criterion for predicting the performance of asphalt pavements to resist common distresses. Lack of bonding can lead to significant asphalt pavement failure. The objective of this study was to investigate the application of zeta potential analysis for the prediction of adhesion between bitumen and stone aggregate based on their surface charge. The adhesion behaviour of four (4) aggregate sources (dolomite, dolerite, andesite, and quartzite) to a 70/100pen grade bitumen was studied. The adhesion of the bituminous binder to stone aggregate was determined with the conventional Rolling Bottle test method used at the CSIR. The zeta potential for macroscopic solid surfaces of aggregates with similar physical properties and bitumen was measured using an electrokinetic analyser at different pH levels. The results predicted that dolomite aggregates had better adhesion when compared to dolerite, andesite, and quartzite aggregates. The Rolling Bottle results are ranked in a way consistent with the zeta potential predictions when the isoelectric point (IEP) is used. The characterization of the aggregate surface chemistry in the zeta potential vs pH curves has provided a better insight into the behaviour of aggregates in different pH conditions. The location of the IEP as per the characterization allows for a better prediction of aggregate-bitumen adhesion behaviour. The content of Fe_2O_3 and CaO present in the aggregates (i.e., dolomite and dolerite) results in better adhesion than the aggregates with a higher content of SiO_2 (i.e., quartzite and andesite). The study shows that the zeta potential analysis has the potential to predict the adhesion of bituminous binder to stone aggregate.

Keywords: Bitumen-Aggregate Adhesion, Isoelectric point (IEP), Rolling Bottle test, Zeta potential.

1. BACKGROUND

About 80% of paved roads in South Africa are constructed using asphalt surfacing. The performance of asphalt pavements is dependent on the behaviour between aggregates and binders that can be affected by moisture damage or water ingress. Moisture damage can lead to pavement distresses that include stripping, ravelling, rutting, bleeding, cracking and potholes (Zhang et al., 2015).

The adhesion is known as the tendency of 'staying together' of two dissimilar materials held together by interfacial forces (ASTM D907, 2015). The adhesion between aggregates and bitumen is an important measure that describes the quality of the asphalt mixture (Paliukaite et al., 2016). The 'staying together' between binder and aggregate in an asphalt mix is the key to preventing the moisture damage that results in stripping. Stripping involves water or water vapour penetrating between the bitumen and aggregates, breaking the adhesive bond between the two components. The current understanding is that the primary factors that affect stripping in asphalt mixtures include chemical and physical properties of both binder and aggregates (Hefer et al, 2005; Zhang et al., 2015; Nageswaran, 2016).

The propensity to the stripping of bituminous binders and stone aggregates is currently evaluated using the chemical immersion test method (TMH1, 1986) and the Rolling Bottle test method (BE-TM-BINDER-7, 2011) method. Although these standard methods supported the current understanding of the influence of adhesion towards asphalt pavement performance, they have several limitations such as being time-consuming and subjective.

Numerous research efforts have been conducted to understand the concept of adhesion and to find the most appropriate test method(s) to determine the affinity between bitumen and aggregates (Paliukaite et al., 2016). There have been many adhesion theories proposed to further understand the mechanism of adhesion: (1) adsorption theory, (2) mechanical theory, (3) electrostatic theory, (4) diffusion theory, (5) weak boundary layer theory (Nageswaran, 2016).

This paper explores the use of a modern solid surface zeta potential analysis using the streaming potential technique for the prediction of adhesion between bitumen and stone aggregate (as per the electrostatic theory). The use of the zeta potential technique to quantify the surface charges of bitumen and aggregates was investigated by Labib et al. (2007). That study used electrophoreses (electrostatic theory) to investigate the proton transfer surface properties of both bitumen and aggregates in an aqueous environment. The authors developed zeta potential test procedures to quantify surface charge characteristics of the material. Surfaces charges of aggregates as a function of pH reveal three (3) distinct surface chemistry regions under different environmental conditions. The study introduced isoelectric points (IEP) as a quantitative parameter that describes surface acidity or basicity of material in aqueous systems. Testing and analysis led Labib et al. (2007) to consider the zeta potential technique as a viable method for predicting adhesion quantitatively.

Using an electrokinetic analyser (SurPASS^{TM}3) device, the surface charge can be determined to give information about surface functionality that can be used to predict compatibility between bitumen and aggregates. The research objective is to improve pavement performance through better predictions of adhesive behaviour between bituminous binders and aggregates, at a fraction of the time used in current test methods.

2. METHODOLOGY

This study focused on testing four (4) aggregate samples and one (1) bituminous binder type (70/100pen grade bitumen) conforming to SANS 4001-BT1 (2016) specification. The physical properties of aggregate samples were determined before commencing the Rolling Bottle test. Representative samples were obtained to conduct the zeta potential test.

Four (4) types of aggregates samples were selected for the study:

- Representative samples were obtained from each aggregate type by riffling.
- 50 particles were randomly selected from each of the aggregate types for the image analyzer scanner to characterize their surface texture (Moaveni, 2015) and flat and elongation ratio (Moaveni, 2015) of particles.
- Flakiness index testing (SANS 3001-AG4, 2015) was also performed on all coarse aggregates samples to investigate the physical properties for classification.

The zeta potential for macroscopic solid surfaces of aggregates and bitumen binder at different pH levels were measured using the SurPASS™ 3 device.

Rolling Bottle testing (BE-TM-BINDER-7-2011, 2011) was performed for each aggregate coated with a 70/100pen grade bitumen to determine the stripping percentage. 500g of aggregate passing 13.2 mm sieve and retained on the 9.5 mm sieve were rinsed, then dried for 1 hour at 163°C. The bitumen was heated to 163°C until soft. 6g of bitumen and 194g of aggregate were mixed well in a one (1) litre glass beaker. The mixture was placed back in the oven at 163°C for 15 minutes, then taken out. The mixture was mixed again for 2 minutes to ensure the aggregates were fully and evenly coated. The coated aggregates were allowed to cool for 24 hours, then placed in a bottle containing 300ml of distilled water. The samples were placed on the Rolling Bottle machine and rolled for 96 hours at ~150 revolutions per minute. The test is concluded after curing for 7 days in distilled water, where the degree of coating is visually evaluated and recorded.

3. APPARATUS DETAILS

The SurPASS™3 device for zeta potential analysis of macroscopic solid surfaces is shown in Figure 1. SurPASS™3 series was used to analyze the samples in this study. The SurPASS™3 instrument is applicable for the zeta potential analysis of solids of almost any shape and sizes like fibres, foils, sheets, textiles, powders or granules. The SurPASS™ 3 instrument is intended for use with diluted aqueous solutions of inorganic salts, acids and bases. The SurPASS™3 series was equipped with integrated processing software that records pH, conductivity, permeability, pressure, resistance, conductivity, temperature streaming current and streaming potential.

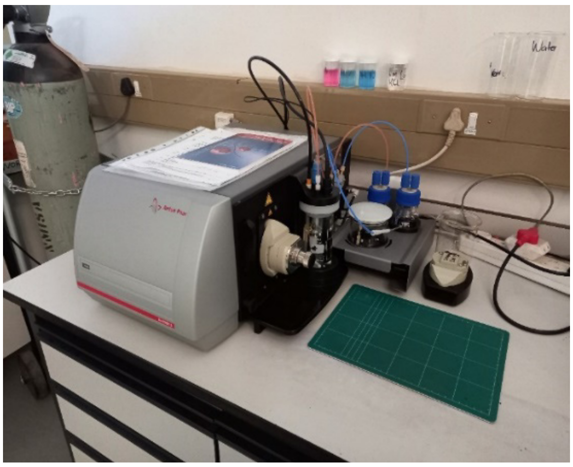

Figure 1: SurPASS™ 3 series (Anton Paar)

3.1 Material Details

3.1.1 Aggregates

The aggregates were sourced from commercial quarries around Gauteng. The material selection used ensured that aggregates with different geological rock formations were selected. The aggregates were andesite, quartzite, dolomite and dolerite. The coarse aggregate samples were prepared by riffling, sieving and washing. The aggregate samples for surface charge analysis were prepared by crushing, washing, drying and sieved to obtain material that is passing the 5 mm sieve and retained on the 0.150 mm sieve size. The sample retained on the 0.150 mm sieve was dried in an oven at 105°C for 1 hour and allowed to cool to room temperature. Approximately 1 ± 0.5 g of sample was weighed by scooping to fill the measure.

3.1.2 Bitumen Binder

A standard 70/100pen grade bitumen (standard binder) was obtained from a commercial asphalt plant in Gauteng. The binder was heated in an oven set at 160°C until fluid, then stirred using a spatula to ensure a homogenous sample. Double-sided tape is placed throughout the Gap Cell surface. This is to ensure easy removal of the bitumen sample after testing and prevent staining of the cell. Approximately 1-2 grams of the bitumen is spread evenly on the cell covered with double-sided tape. This is to ensure that the bitumen does not form lumps and is tested as a thin layer in the cell (SurPASS^{TM}3 reference guide, 2019).

4. ZETA POTENTIAL SURFACE MEASUREMENTS DETAILS

The zeta potential for aggregates and bitumen binder was measured using an electrokinetic analyser (SurPASS^{TM}3 series, Anton Paar). The surface potential was determined as a function of pH in a 0.001 Potassium chloride (KCl) electrolyte solution. The varying of the solution pH was done by addition of 0.05 mol/l of sodium hydroxide (NaOH) solution prepared by dissolving a 0.5 ± 0.1 g of NaOH in 250 ml of deionised or ultra-pure water. A 0.05 mol/l of hydrochloric acid (HCl) solution was prepared by mixing 1.21 moles of HCl of 32% purity in 250 ml of deionised or ultra-pure water through the instrument automatic titration unit. Four measurements for zeta potential were carried out at each pH point.

The zeta potential reflects the charging behaviour at a solid-liquid interface that can be generated by either acid-base reactions of functional groups and/or the adsorption of ions. When the zeta potential is determined by the measurement of an electrokinetic effect generated by a tangential flow of liquid across a solid surface, the method is referred to as the streaming potential technique (Luxbacher, 2014).

For granular material, the zeta potential (ζ) is obtained from a derivative of the Helmholtz-Smoluchowski equation:

$$\zeta = \frac{dU_{\mathrm{str}}}{d\Delta p} \times \frac{\eta}{\varepsilon \times \varepsilon_0} \times \mathrm{K}_B \qquad (1)$$

dUstr/dΔp is the slope of streaming potential vs differential pressure, η and ε are water viscosity and dielectric coefficient, ε_0 is the vacuum permittivity, and κ_B is the electrolyte conductivity.

Streaming potential measurements were conducted using a cylindrical cell filled with an aggregate sample that has been mounted between support disks and filters (with 25 µm mesh) on both sides of the granular sample plug. An electrolyte solution was passed through the aggregate sample. The aggregate samples were cautiously washed with sodium chloride (NaCl) electrolyte solution to exclude particle sizes less than 25 µm before conducting the test. The permeability index was monitored and adjusted to fit a range of 85 – 115 to acquire accurate zeta-potential readings. A pressure range of 100 mbar – 400 mbar was then applied between both ends of the aggregate sample plug. The bitumen sample was measured using a gap cell and followed a similar procedure as used for aggregates samples, with the same electrolyte solution.

The equipment pH electrode was calibrated at three buffer standard solutions of known pH value (pH 4, pH 7 and pH 10). The conductivity electrode was calibrated using a 0.1 mol/l KCl solution or a conductivity standard solution.

5. RESULTS AND DISCUSSION

5.1 Aggregate Physical Properties for the Rolling Bottle Test

The aggregates were tested for shape and texture properties prior to the Rolling Bottle testing. The results indicate that the silicious aggregates selected had mostly similar shape and texture properties to test along with the dolomite (see Table 1).

Table 1: Physical properties of aggregates for the Rolling Bottle test

Property	Test Results				Test Method
	Dolomite	Dolerite	Quartzite	Andesite	
Flat & elongation ratio	3.0	3.3	1.7	1.6	Moaveni, 2015
Surface texture	3.6	5.7	6.7	5.3	
Flakiness index	12	19	17	18	SANS 3001- AG4, 2015

5.2 Adhesion of Bitumen – Test Results

The adhesion of bituminous binder to stone aggregates was determined according to the Rolling Bottle test method (BE-TM-Binder-7-2011, 2011). The results based on a visual analysis are reported in Figure 2. The data shows that dolomite aggregates had a bitumen coating of 27% remaining after the test. This was the highest percentage of coating observed for the tested samples. The andesite and quartzite had 5% bitumen coating remaining.

Although Paliukaite et al. (2016) found that the Rolling Bottle test method was the most suitable test for determining an affinity between bitumen and aggregate samples, the visually based approach was considered as a major disadvantage - the evaluation is inherently subjective tand cannot accurately quantify the percentage coating of the aggregate. The researchers report that the most stable results were determined after 6 hours of rolling.

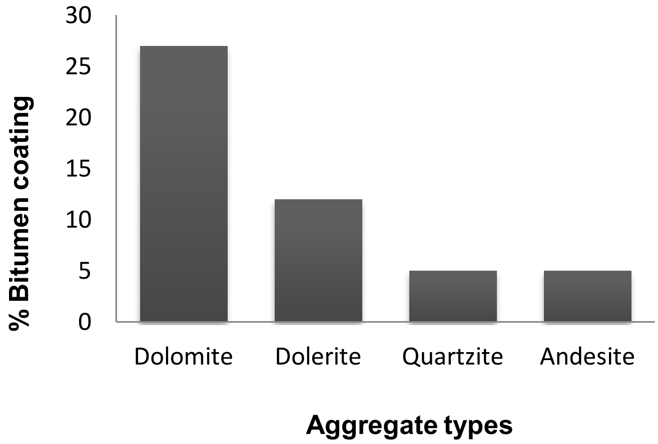

Figure 2: Rolling Bottle results

5.3 Zeta Potential Results

Electrokinetic techniques enable the measurement of the zeta potential of particles as a function of the pH of the electrolyte. A representation of the zeta potential vs pH curve with three separate pH regions that characterize the aggregate surface chemistry under different aqueous conditions is shown in Figure 3. Labib et al. (2007) established a detailed, fundamental relationship between zeta potential, isoelectric point (IEP) and acid-base proton transfer properties of aggregate surfaces. These pH regions are discussed in detail for siliceous and calcareous aggregates in Table 2.

Table 2: A detailed description of pH regions that characterize the aggregate surface chemistry (Labib et al., 2007)

pH regions	Siliceous aggregates	Calcareous aggregates
Region I	• Adsorption of protons results in a positive surface charge indicated by positive zeta potential. • No dissolution of the silica surface is expected.	• The positive charge on surfaces is attributed to proton adsorption and calcium ion dissolution. • The dissolution of the surface layer is expected in the presence of mineral or carboxylic acids. • Acid conditions enhance stripping of the surface layer, promoting a cohesive failure.
Region II	• Presence of IEP; equilibrium adsorption of protons and hydroxyl ions formation. No dissolution is expected to take place.	• The IEP is found in this region, confirming the dominant basic character of the surfaces in aqueous conditions.
Region III	• Dominated by hydroxyl formation. • Dissolution of the silica surface. • The rate of dissolution depends on pH, ion composition, organic compounds and temperature.	• Formation and dissociation of surface hydroxyl groups. • No significant solubility is expected for limestone aggregates.

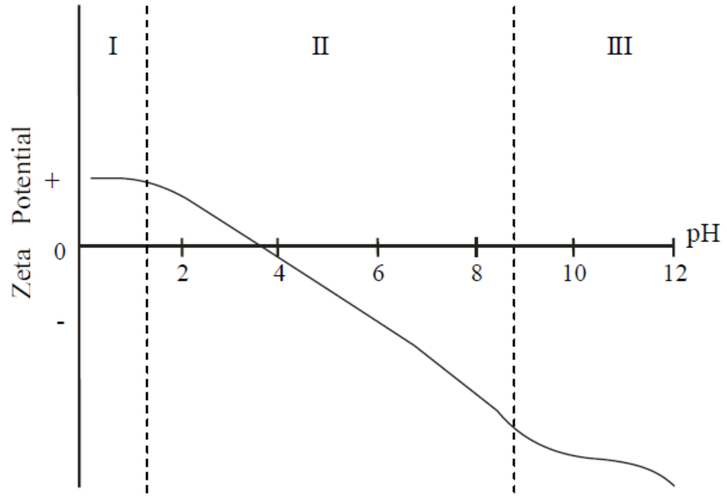

Figure 3: A characterization of aggregate surface chemistry pH regions (Labib et al., 2007)

For this study, the zeta potential was performed on the aggregate and bitumen samples at various pH levels. The relationship between zeta potential and pH of the aggregate is shown in Figure 4, whereas that of the bitumen is shown in Figure 5. The aggregates showed a decreasing trend in zeta potential value with an increase in pH. Dolomite and dolerite also exhibited changes in zeta potential from positive to negative with increasing pH. According to Jusang et al. (2011), a higher alkaline oxide or silica content results in more surface charges. Given bitumen is slightly acidic, the results were analysed at pH 6-7 where they predicted the following poor adhesion ranking: dolomite>andesite>dolerite>quartzite.

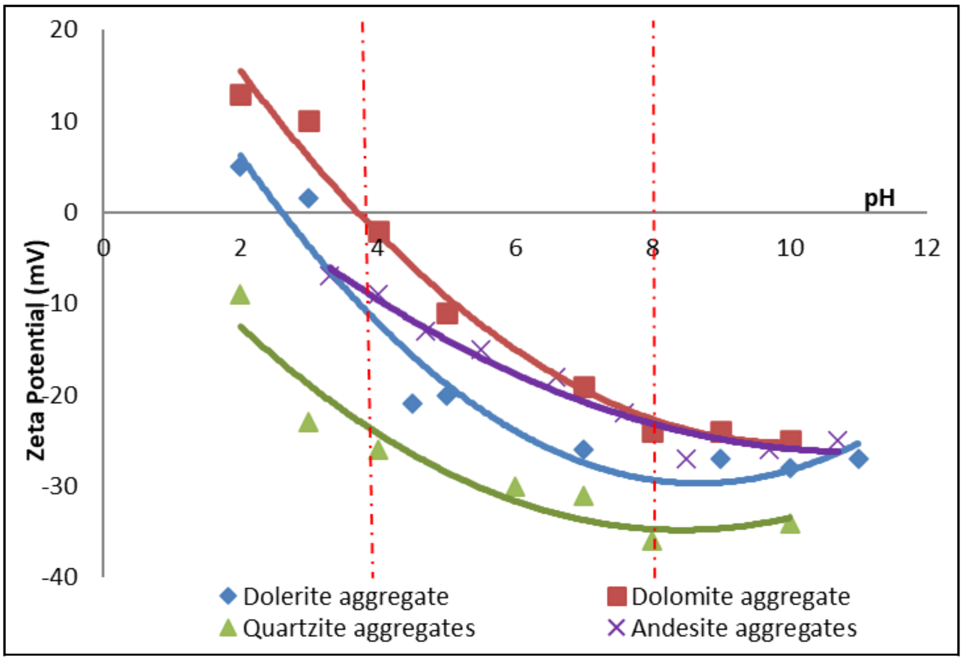

Figure 4: Zeta potential of each aggregate at different pH solutions

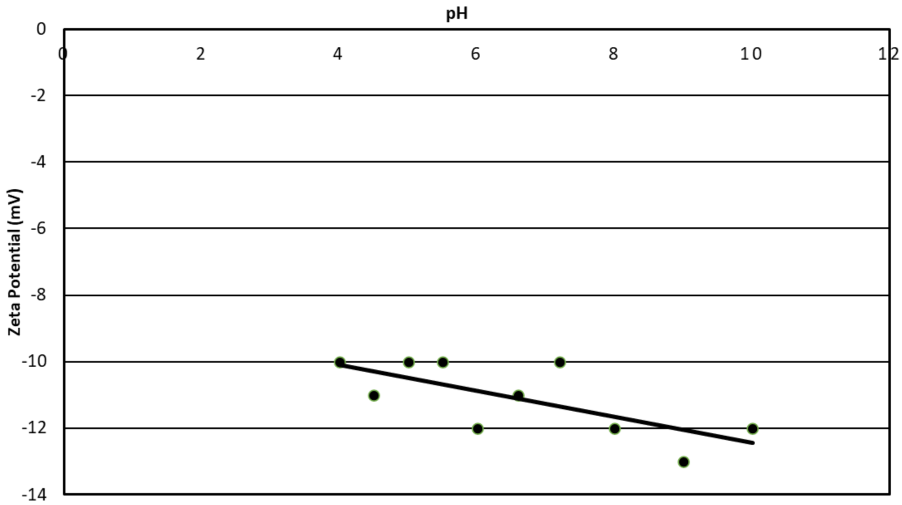

Figure 5: Zeta potential at various pH for a 70/100 penetration grade bitumen

The aggregate surface chemistry behaviour is consistent with the zeta potential vs pH curve and pH regions shown by Labib et al. (2007), see Figure 6. This shows consistency in the test method and further validates the use of the zeta potential test in predicting surface charge behaviour.

In Figure 6, Labib et al. (2007) characterized the dominating surface charges of aggregates and bitumen in aqueous environments. Siliceous aggregates, granite labelled RJ, displayed a mostly negatively charged surface over a wide pH range compared to the calcareous aggregates (limestone labelled RD) that had a mostly positively charged surface. Bitumen was also observed to be mostly negatively charged across a wide pH range. The negatively charged granite resulted in poor adhesion with the bitumen (like charges), thereby promoting stripping. On the other hand, the positively charged limestone showed better adhesion with the bitumen due to their opposite surface charges. This promotes attraction and a stronger adhesive bond. These observations are consistent with results displayed in figure 5 and 6. Dolomite (calcareous) displayed a positive charge over a larger pH range compared to the siliceous aggregates that were tested. Dolomite was observed to have a stronger adhesive bond with the negatively charged bitumen and this was substantiated by the Rolling Bottle test results. The work done by Labib et al., 2007 is consistent with the results of this study.

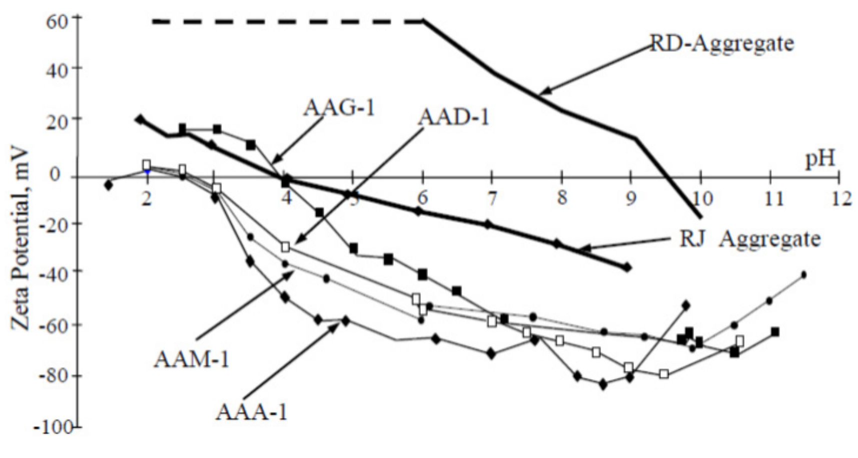

Figure 6: Zeta potential results for siliceous and calcareous aggregates with different bitumen types (Labib et al., 2007)

5.4 Correlating Zeta Potential Results with Adhesion

This study analysed the isoelectric point (IEP) of aggregate samples from the zeta potential results for correlating with bitumen adhesion. An IEP is a pH value at which the zeta potential is zero. The Hefer (2004) dissertation stated that bitumen conventionally has a net acidic character, where function groups such as carboxylic acids will act as proton donors. These proton donors tend to form more durable bonds with strong proton-accepting aggregate surfaces. Figure 7 illustrates those calcareous aggregates such as limestone have an increased proton accepticity at higher IEP compared to siliceous aggregates. This results in calcareous aggregates forming stronger bonds (adhesion) with bitumen.

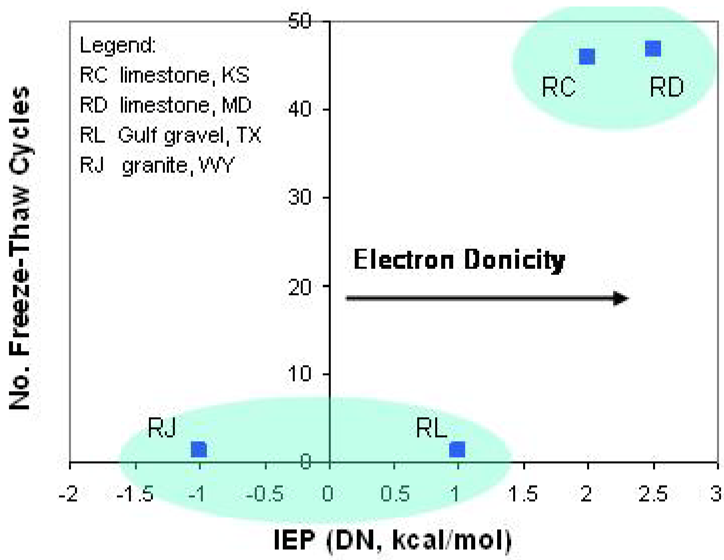

Figure 7: Proton accepticity between different aggregates. Bitumen-aggregate performance linked to the isoelectric point (IEP) on the pH scale (Hefer, 2004)

The electric double-layer theory allows the application of electrokinetic properties to calculate the free energy of electrostatic interaction at different pH levels. In this study, the zeta potential data was used to quantify and evaluate the significance of this interaction term in bitumen-aggregate interactions. The findings demonstrated that electrostatic interactions in bitumen-aggregate electrolytes are generally repulsive at equilibrium pH conditions, but that a decrease in pH can ultimately lead to attraction between these surfaces (Labib et al., 2007). Given the limitation of zeta potential results at a given electrolyte composition (i.e., at a certain pH), the isoelectric point (IEP), where $\zeta = 0$ mV, was determined from the equations of the lines and predicted the following adhesion ranking: dolomite> dolerite>andesite>quartzite. The dolomite sample reaches IEP at the highest pH of 3.7 (Figure 8) IEP rankings also show that dolerite exhibited the second strongest bond with bitumen where its IEP was at a pH of 2.6. This agrees with the Hefer (2004) dissertation that reported aggregates that have IEP at higher pH form the strongest bond with bitumen because they have a high proton accepticity. The comparison of zeta potential results with the Rolling Bottle test results in Table 3 indicates an agreement that less binder stripping with dolomite (better adhesion) was expected when compared to the others. However, the rest of the order of Rolling Bottle adhesion results ranks as follows: dolomite>dolerite>andesite/quartzite.

This indicates that the Rolling Bottle test results needed more than zeta potential characterisation at a specific pH to predict adhesion. A more improved ranking was observed with the IEP, and a fair correlation between the IEP and the Rolling Bottle results is shown in Figure 9 (R^2=0.672). Interestingly, without the quartzite result, the IEP correlates almost perfectly with the Rolling Bottle results as illustrated in Figure 10 (R^2=0.996). Given the limitation of zeta potential results at a given electrolyte composition (i.e., at a certain pH), the isoelectric point (IEP), where $\zeta = 0$ mV, was determined from the equations of the lines of best fit through interpolation and extrapolations, and predicted the following adhesion ranking: dolomite> dolerite>andesite>quartzite.

Júnior et al. (2019) cited a study by McCann et al. (2005) on the effect of the chemical composition of the aggregate on asphalt film coating dislocation. The study utilized the Freeze–Thaw Pedestal test to assess how the physical and chemical properties of aggregates impact the moisture-damage resistance. Their findings indicated that the higher the content of iron (Fe_2O_3) and/or calcium oxides (CaO), and the lower the content of silicon (SiO_2) and potassium oxide (K_2O) in the aggregate, the more resistant to moisture-damage the asphalt mixes become. Aggregates with higher K_2O and SiO_2 percentages (~ greater than 55%) showed a smaller percentage of bitumen coating (poor adhesion). Conversely, aggregates with higher Fe_2O_3 and CaO contents showed a higher percentage of bitumen coating (good adhesion). Quartzite and Andesite contain SiO_2 greater than 55%, with Dolerite only consisting of 45%-55% SiO_2. Dolomite is composed of CaO minerals. The strong correlation between the CaO content and adhesion can validate the use of the zeta potential method to predict adhesion because lime has been proven to be an adhesion promoter additive (acting to prevent moisture damage). The zeta potential method validates studies on lime used in improving adhesion.

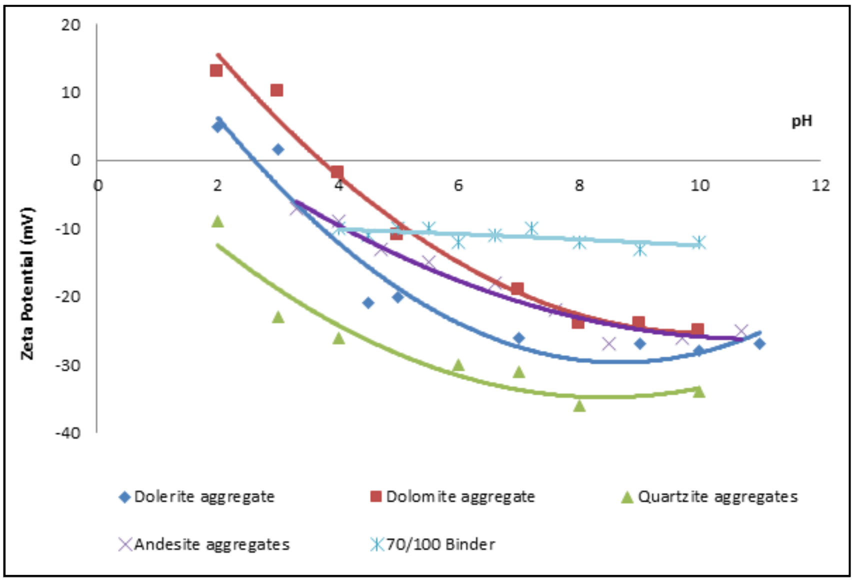

Figure 8: Aggregates and binder zeta potential curves

Table 3: Adhesion ranking of various aggregates based on the zeta potential and Rolling Bottle test

Aggregate	Zeta potential ranking at pH 6-7	Zeta potential ranking at IEP	Rolling Bottle ranking
Dolomite	1	1	1
Dolerite	3	2	2
Andesite	2	3	3
Quartzite	4	4	3

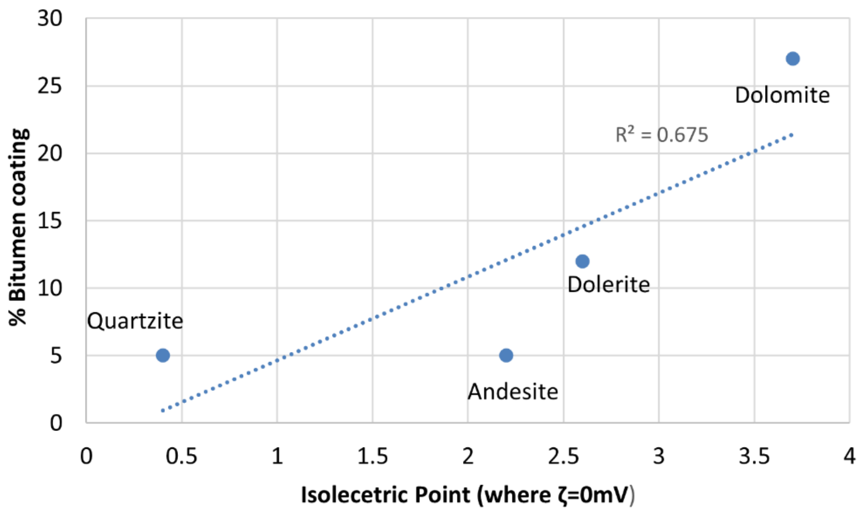

Figure 9: A correlation of the Rolling Bottle adhesion results with the IEP values of the aggregates

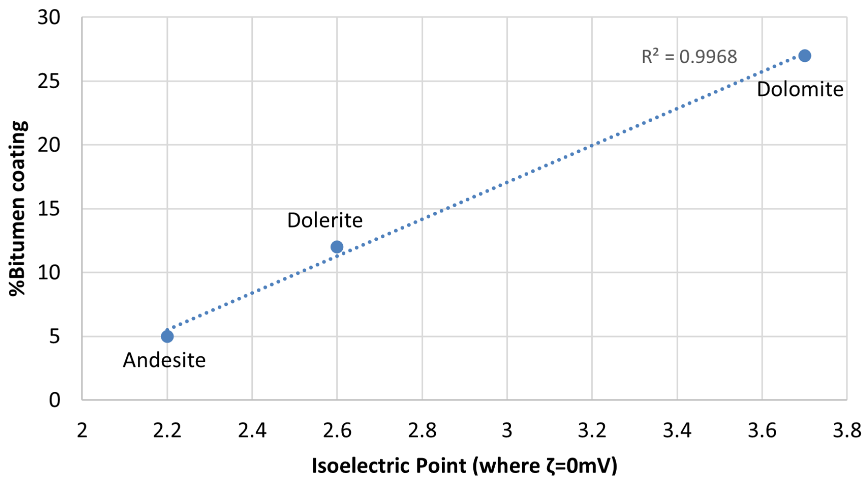

Figure 10: A correlation of the Rolling Bottle adhesion results with the IEP values of the aggregates (without the quartzite result)

6. CONCLUSIONS

This study investigated the effect of surface charge of aggregates and bitumen for predicting aggregate-bitumen adhesion. Understanding the surface charge behaviour of aggregates and the pH value of the IEP can indicate the aggregate-bitumen adhesion behaviour. Previous studies in the field have shown consistency with the results obtained in this study, proving that the zeta potential test can be a viable and accurate method for predicting aggregate-bitumen adhesion. Therefore, the SurPASS^{TM}3 Zeta Potential Analyzer can be further evaluated for consideration as a substitute for the Rolling Bottle test to assess aggregate-binder adhesion behaviour. The effect of aggregate crushing for the zeta potential test may require further investigation. The crushing exposes fresh surfaces that might have different zeta potential characteristics from the original sample used for the Rolling Bottle test.

Although the aggregates for the Rolling Bottle test had mostly similar physical properties, they showed different adhesion behaviour with bitumen. This proves that the surface charge behaviour of aggregates in different pH conditions is vital in predicting bitumen-aggregate adhesion and can verify results from the Rolling Bottle test. This is substantiated by the observation that the chemical composition of aggregates also influences the aggregate-binder adhesion, with aggregates having a higher content of Fe_2O_3 and CaO exhibiting better adhesion than aggregates with higher SiO_2 content. The zeta potential test has the potential to act as an alternative to conventional adhesion tests with substantially shorter turnaround times pending further investigation.

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