Title: THE NET ADSORPTION TEST FOR CHIP SEALING AGGREGATES AND BINDERS

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THE SHRP NET ADSORPTION TEST : APPLICATION TO IRISH CHIP-SEALING AGGREGATES AND BINDERS.

ABSTRACT

The Net Adsorption Test (M-001) developed for the SUPERPAVE mix design procedure is of interest to those concerned with the selection of binders and chippings for chip seals (surface dressings). In this paper, the relevance of the Net Adsorption Test (NAT), which is performed on the dust fraction, for assessing the adhesion performance of chipping sizes (14 mm) used for chip seals and the behaviour of bitumen emulsions is evaluated.

Since the surface chemical composition of 14mm chippings was not found to be statistically different from the composition of the dust from the crushed chippings it was accepted that NAT results were indicative of the adhesion performance of the chippings with the binder used in the test.

In testing bitumen emulsions the prior removal of the water phase by evaporation was necessary.

Results obtained with aggregate/bitumen combinations used for chip sealing in Ireland agreed with the SHRP findings that aggregate type has a dominating influence on binder-aggregate adhesion. However, with aggregate/emulsion combinations the emulsion source had a major effect and the influence and type of emulsion surfactant

was assumed to be responsible for the very specific affinity of these binders for aggregates. This is consistent with results of SHRP studies on the effect of antistripping agents on bonding energies.

If the percent net adsorption is determined on the basis of the total binder in the test solution an overall expression of the binder-aggregate affinity and resistance to moisture damage is provided.

THE SHRP NET ADSORPTION TEST : APPLICATION TO IRISH CHIP-SEALING AGGREGATES AND BINDERS.

INTRODUCTION

The Strategic Highway Research Program's (SHRP) Net Adsorption Test is based on the physical chemical adsorption of a solute (bitumen) from a solution onto a solid (road aggregate). The test provides a fundamental quantitative measure of the affinity between bitumen and aggregate and a means of measuring quantitatively the effects of factors such as moisture, bitumen additives, etc. on the bond.

Previous research, since 1950, has indicated the importance of the influence of aggregate type and properties on the aggregate/binder adhesion bond. Hallberg(1) conducted experiments, from 1950 - 1958, on the influence of aggregate petrography on the aggregate/binder adhesion bond and showed statistically that the adhesion performance of the bond was better with basic (low silica content) as opposed to acidic (silica content >66 percent) rocks.

The SHRP study(2) shows that the mechanism of stripping is failure within the aggregate(3) and not separation of binder and aggregate at the interface. This is due to dissolution, particularly of silica which is relatively soluble at high pH (<9) levels(4). A series of Net Adsorption Tests (NAT) on eleven aggregates and three bitumens confirmed that the aggregate type has a greater influence on adhesion than variations in bitumen type. Each bitumen exhibited high and low levels of adsorption, for example, high adsorption with limestone and low adsorption with granite, but the magnitude of the differences among the aggregates for each bitumen was quite large.

A routine NAT procedure was developed as a preliminary screening method (M-001) for aggregate/binder combinations in the SHRP SUPERPAVE mix design method[5]. If this can be used to evaluate aggregate/binder combinations for chip sealing operations, it would be of particular value in European countries where chip-sealing (surface dressing) is a major road maintenance procedure. The purpose of this paper is to describe the results of an investigation involving aggregates and binders used in chip-sealing in Ireland.

ADSORPTION ISOTHERMS

The Net Adsorption test, as previously mentioned, is based upon the phenomenon of adsorption and in the SHRP investigation liquid adsorption isotherms were studied, as shown in Figure 1. The figure shows the influence of aggregate type on adsorption of bitumen over a range of bitumen solution concentrations.

Adsorption studies were also used to assess the adsorption affinity of various bitumen components. For example, compounds with polar functional groups, (sulphoxides, carboxylic acids and nitrogen bases) were found to be more adsorptive and formed much stronger adhesion bonds than less-polar compound types (ketones and non-basic nitrogen groups). However, desorption studies showed that sulphoxides and carboxylic acids were most susceptible to stripping whereas the ketones and basic nitrogen groups were most resistant[2].

OBJECTIVES

The purpose of this investigation was to determine if the net adsorption procedure could be used to assess the affinity of aggregate/binder combinations for chip sealing.

The M-001 procedure uses the fine aggregate, fraction 4.75 - 0.0 mm, (hereinafter referred to as "dust") of a hot-mix aggregate grading. However, in chip-sealing only single sized aggregates are used, usually 10 and 14 mm sizes, though even 16 mm or larger sizes are used in some circumstances. The net adsorption test is not practical with the aggregate sizes used in chip sealing since to maintain the same ratio of solvent volume to aggregate used in the research investigation a large quantity of the solvent would be required.

In addition the method must be applicable to the most common type of surface dressing binder which is bitumen emulsion.

The objectives therefore were:

(i) to determine if results obtained on the dust fraction are applicable to the performance of larger size chippings.

(ii) to evaluate bitumen emulsion binders by the NAT procedure.

CALCULATIONS AND EVALUATION.

The NAT determines:

(i) The affinity between bitumen and aggregate - Initial adsorption.

(ii) The moisture sensitivity of the aggregate/binder bond - Net Adsorption or the amount of bitumen remaining on the aggregate after water is added.

In order to calculate both the initial and net adsorption three measurements on the solution of bitumen in the solvent (toluene) are carried out:

(i) Initial concentration of bitumen/toluene solution, A_1.

(ii) Solution concentration after 6 hours in contact with the aggregate, A_2.

(iii) Solution concentration after addition of water to the aggregate/bitumen solution, A_3.

The solution concentrations are determined by a spectrophotometer technique at 410nm.

The Initial Adsorption is given by : $A_i = \dfrac{VC(A_1 - A_2)}{WA_1}$

where V = volume of solution - 140ml

C = concentration of bitumen/toluene solution.

A_1/A_2 = Solution concentration measurements.

W = weight of aggregate sample to nearest 0.001g.

The Net Adsorption is given by : $A_n = \frac{VC(A_1 - A_3)}{WA_1}$

where volume at this stage = 136ml.

Percent Net Adsorption = $\frac{A_n}{A_i} \times 100$

These calculations are used in the standard procedure (M-001) and criteria for performance were suggested[2] as shown in Table 1.

The authors found that the precision of the method was excellent using the graded dust fraction shown in Table 2. This is the grading used in the SHRP research investigations and unlike the grading used in M-001 it contains no passing 75μm fraction. Otherwise the fractions are in proportion with the standard asphalt concrete grading ASTM D3515. The use of a standard grading minimises variations in surface area which SHRP showed to have a major influence on the results of the test. On repeat testing of a number of aggregate/binder combinations the standard deviation was <0.05 mg/g compared to the value of 0.08 mg/g as reported SHRP. All results presented in this paper are the means of measurements carried out in triplicate.

Expressing the results, as the percentage net adsorption, although effective in illustrating the moisture sensitivity of the bond, does not take into account differences in the amount of bitumen initially adsorbed by the aggregate. For example, in Table 3 of the two aggregates tested with binder 1, aggregate A has a net adsorption value of 71.3 percent and aggregate B a value of 80.8 percent. This suggests that both these values are acceptable (see Table 1). However, if these results are re-evaluated[6], as suggested by Woodside et al., to express the initial and net adsorption as a percentage of the total bitumen in the solution, a more discriminating assessment of affinity and resistance to stripping is possible. On re-evaluation, it is apparent that aggregate B actually has a lower initial adsorption, 42.7 per cent, than A, 48.2 per cent, and it has only a marginally better net percent adsorption value than A (35.7 - 35.3).

The performance criteria in Table 1 are not applicable to the re-evaluated data and ranges of values associated with acceptable marginal and poor adhesion performance and resistance to stripping are unavailable at this stage. These need to be developed in the light of the known performance of aggregates and binders.

EXPERIMENTAL WORK

Effect of Aggregate Size:

Stepwise regression of the SHRP results[(2)] indicated that the chemical and physical properties of the aggregate have a major influence on the net adsorption of the test results. These factors are listed in decreasing order of impact in Table 4.

It was decided therefore, that analysis of the chemical composition of the bulk dust fraction and the surface of 14mm chippings could provide a means of determining if NAT results (carried out on the dust fraction) are acceptable for assessing the performance of larger aggregate sizes. Accordingly measurements of the chemical composition of the surface (two faces) of the 14mm sized aggregate were performed by an energy dispersion technique after which the aggregate particle was crushed to passing 100 μm and analysed by X-Ray Fluorescence Spectroscopy. The elemental composition of the surface of the 14mm chippings and the bulk composition of the dust, obtained on crushing the chippings, are compared for all seven aggregates in Figure 2.

With the exception of silica (SiO_2) the composition of the surfacing of the chippings and the bulk composition of the dust were similar and varied only by the order of 2-4 percent. Though the silica contents varied by the order of 5-10 percent a t-test comparison for correlated samples showed that these differences are insignificant at a level of $p<0.01$. Based on the hypothesis that the means of the sample results i.e. 14mm size and crushed dust, are the same, $\mu_1 = \mu_2$ or $\mu_1 - \mu_2 = 0$, the significance of these differences was determined by comparing the calculated t value (from the results) with a critical t value at a particular significance level. These calculations are illustrated in Table 5. In view of this finding and the strong influence of chemical composition of the aggregate on NAT results, it was accepted that results carried out on the dust fraction can be used as an adhesion performance indicator of the larger, 10-14mm size, chippings with the binder used in the test.

Testing using Bitumen Emulsions:

In testing bitumen emulsions it is first necessary to remove the water from the solid residue (bitumen containing emulsifying agent). This was achieved by the controlled evaporation of the water from the emulsion in an apparatus in which the emulsion is left to 'cure' in a stream of air under constant pressure and constant temperature for 18 hours[7]. This was sufficient to isolate the bitumen and the emulsifying agent for use in the Net Adsorption test.

VALUES OBTAINED ON IRISH CHIP SEALING AGGREGATES AND BINDERS.

Seven Irish aggregates were selected for the test programme. These are typically used for chip sealing in Ireland and they comprised igneous, metamorphic and sedimentary categories of rock as shown in Table 6.

Binders were chosen from five different Irish suppliers : two paving grade bitumens (100 pen.) and three cationic bitumen emulsions.

Paving grade bitumens:

Table 7 and Figure 3 illustrate the results obtained for four of the aggregates with bitumens 1 and 2.

The percent net adsorption values range from 75.5 percent (schist A) to 86.8 percent (gritstone) with bitumen 1 and from 77.3 percent (schist A) to 83.5 percent (schist B) with bitumen 2. Of the four aggregates, schist A appears to have the lowest stripping resistance with both binders. The differences in net adsorption for these aggregates with bitumen 1 are quite large; there is an 11 percent difference between the result obtained with schist A and gritstone and a 6 percent difference between schist B and gritstone.

An interaction diagram (Figure 4) indicates that the influence of binder type varies according to the type of aggregate. In the case of granite and gritstone, bitumen 2 has an adverse effect on the net adsorption value indicating a greater suspectibility to stripping. With schist A and B, however, bitumen 2 has a positive effect on the net adsorption value, indicating a superior stripping resistance. Thus while the aggregate properties play a very significant role in determining the strength and durability of the bond, the type of binder can also have an important effect.

The results given by bitumen 2, with granite and gritstone in particular, are quite similar. However, re-evaluation of the results, as described previously, to express the initial and net adsorption as a percentage of the total bitumen in the solution shows (Table 8) that the adsorption behaviour varies quite considerably. It is clear that bitumen 2 has a greater affinity for granite with an initial adsorption of 45.7 percent compared with 37.1 percent for gritstone. The net adsorption values, of 37.1 and 30.5 percent respectively, indicate that the gritstone has a marginally higher stripping resistance than the granite.

Bitumen Emulsions:

Table 9 presents the results obtained for the seven aggregates with the three cationic bitumen emulsions. The results are calculated according to both the SHRP and Woodside methods. Figure 5 illustrates the NAT results in bar-chart form.

The source of emulsions appears to play a more significant role in the effectivenness of the adhesion bond than do the variations in the source of paving grade bitumens. For example, in the case of emulsion 1, basalt has a net adsorption value of 66.5 percent, limestone has a value of 90.1 percent and schist A a value of 75.9 percent. Comparison of Table 9 with Table 7 shows that some values are lower than those obtained with paving grade bitumens and some aggregate/emulsion combinations are actually below the acceptable limits of 70 per cent recommended by SHRP. Granite, with bitumen 1 and 2 has high net adsorption values of 83 and 79.4 percent respectively but with emulsions 1 and 2 substantially lower values, 63.8 and 64.3 percent, were obtained. Similar effects were observed with the gritstone in particular, with a 15 percent difference between bitumen 1 and emulsion 2. In some cases, therefore the emulsion type can have an adverse effect on the moisture sensitivity of the bond. The affinity of an aggregate and bitumen with surfactant appears to be unique for the type of surfactant and aggregate and SHRP investigations on bitumens modified with anti-stripping agents provided similar results, as shown in Figure 6.

The re-evaluated initial and net values, Table 9 and Figure 7, indicate that granite/emulsion combinations have the lowest affinity of all combinations of aggregate type and emulsion source. Initial and net adsorption values are 38.9 and 25.7 percent respectively with emulsion 1, 40.5 and 26.8 percent with emulsion 2 and 37.1 and 30.9 per cent with emulsion 3.

CONCLUSIONS

1. The Net Adsorption Test (M-001) developed for the SUPERPAVE procedure was

used to rank the affinity of Irish aggregate/binder combinations manufacture and for chip seals (surface dressings).

2. The chemical composition of the surface of 14mm chippings from seven different sources was not statistically different from the chemical content of the dust obtained on crushing the chippings and on this basis it was accepted that NAT results were indicative of the adhesion performance of surface dressing chippings when applied with the binder used in the test.

3. The procedure used for carrying out the test with bitumen emulsion binders is to first remove the water phase by evaporation so that the binder in the solvent comprises the bitumen with the surfactant.

4. The results obtained with aggregate/bitumen combinations confirm the SHRP findings that the aggregate type has a dominating influence on aggregate/binder adhesion. However, in testing aggregate/emulsion combinations the test showed that the emulsion source had a major effect and the presence of the surfactant may be responsible for the specific affinity of these binders for particular aggregate types as shown by the examples:

Aggregate	Emulsion	Net Adsorption (Percent)
Limestone	Emulsion 1	90.1
	Emulsion 2	90.6
	Emulsion 3	77.2
Granite	Emulsion 1	63.8
	Emulsion 2	64.3
	Emulsion 3	80.5
Sandstone	Emulsion 1	87.9
	Emulsion 2	81.0
	Emulsion 3	70.4

This finding is consistent with results of SHRP studies in which the effect of antistripping agents on bonding energies were investigated.

5. The procedure in method M-001 of expressing the net adsorption as a percentage of the initial adsorption fails to take into account differences in the initial adsorption. To rectify this omission consideration should be given to reporting the percentage net adsorption of the total bitumen in the solution, as proposed by Woodside. Performance criteria for the re-evaluated data need to be developed.

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TABLE 1 Criteria suggested (SHRP) for Aggregate/Binder Adhesion Performance

Percent Net Adsorption	Aggregate/binder bond performance
>70	Good
55-70	Marginal
<55	Poor

TABLE 2 Grading used for the Net Adsorption Test

Sieve Size	Percent Retained	Weight Retained (g)
2.36 mm	8.0	4.3
1.18 mm	25.0	13.5
600 μm	17.0	9.1
300 μm	23.0	12.4
150 μm	14.0	7.5
75 μm	6.0	3.2
		Total 50

TABLE 3 Recalculation of NAT Results as Suggested by Woodside et al

Calculated Net Adsorption according to SHRP		Re-evaluated adsorption according to Woodside et al[6]	
	Percent	Initial percent	Net percent
Aggregate A	71.3	48.2	35.3
Aggregate B	80.8	42.7	35.7
Performance Criteria -		Not Available	
Acceptable	>70		
Marginal	55-70		
Poor	<55		

TABLE 4 The Influence of Aggregate Properties on Net Adsorption[2]

Aggregate Variables	Correlation Coefficient
Potassium Oxide	0.48
Surface Area	0.71
Calcium Oxide	0.75
Zeta potential	0.87
Sodium Oxide	0.90

TABLE 5 Statistical t-test Analysis of the Silica, Alumina and Iron Content of Dust and 14mm Sizes of the Selected Aggregates

Silica	Dust	14 mm	t-Test: Paired Two-Sample for Means	*Dust*	*14 mm*
Basalt	51.1	41.7	Mean	59.75714	52.1
Granite	68.2	63.4	Variance	275.4162	180.5833
Gritstone	65.7	63	Observations	7	7
Limestone	30.8	30.9	Pearson Correlation	0.947792	
Sandstone	84.8	67.7	Pooled Variance	211.3717	
Schist A	60.2	51.9	Hypothesized Mean Difference	0	
Schist B	57.5	46.1	df	6	
			t	**3.51301**	
			P(T<=t) one-tail	0.006313	
			t Critical one-tail	3.142668	
			P(T<=t) two-tail	0.012625	
			t Critical two-tail	**3.70743**	

Alumina	Dust	14 mm	t-Test: Paired Two-Sample for Means	*Dust*	*14 mm*
Basalt	19.2	18.85	Mean	12.02	14.85429
Granite	14.4	12.2	Variance	24.82053	19.65056
Gritstone	13.5	13.2	Observations	7	7
Limestone	4.16	9.05	Pearson Correlation	0.700401	
Sandstone	6.98	11.11	Pooled Variance	15.4682	
Schist A	13.5	17.95	Hypothesized Mean Difference	0	
Schist B	12.4	21	df	6	
			t	**-2.0383**	
			P(T<=t) one-tail	0.043825	
			t Critical one-tail	3.142668	
			P(T<=t) two-tail	0.08765	
			t Critical two-tail	**3.70743**	

Iron	Dust	14 mm	t-Test: Paired Two-Sample for Means	*Dust*	*14 mm*
Basalt	8.93	14	Mean	4.991429	6.582857
Granite	5.09	6.12	Variance	5.451381	12.85202
Gritstone	5.62	5.65	Observations	7	7
Limestone	1.65	2.93	Pearson Correlation	0.93818	
Sandstone	2.72	3.9	Pooled Variance	7.852812	
Schist A	5.56	7.2	Hypothesized Mean Difference	0	
Schist B	5.37	6.28	df	6	
			t	**-2.6124**	
			P(T<=t) one-tail	0.019997	
			t Critical one-tail	3.142668	
			P(T<=t) two-tail	0.039993	
			t Critical two-tail	**3.70743**	

TABLE 6 Mechanical and Physical Properties of Aggregates Selected for the Test Programme

Aggregate	Class	PSV	AAV	ACV	% Water Absorption	Specific Gravity
Basalt	Igneous	55	3.0	15	1.0	2.73
Granite	Igneous	52	3.3	26	0.5	2.69
Gritstone	Sedimentary	65	7.0	17	0.7	2.69
Limestone	Sedimentary	62	8.4	18	0.7	2.70
Sandstone	Sedimentary	63	5.3	20	1.64	2.54
Shist A	Metamorphic	63	7.8	16	1.09	2.70
Schist B	Metamorphic	62	8.1	16	0.7	2.69

TABLE 7 Percent NAT results obtained with Irish Aggregate and Bitumens

Aggregate	Bitumen 1			Bitumen 2		
	A_i Initial Adsorption mg/g	A_n Net Adsorption mg/g	% NA	A_i Initial Adsorption mg/g	A_n Net Adsorption mg/g	% NA
Granite	1.16±0.02	0.96±0.02	**83.0**	1.28±0.05	1.01±0.01	**79.4**
Gritstone	1.14±0.02	0.99±0.01	**86.8**	1.04±0.02	0.83±0.02	**79.8**
Schist A	1.36±0.05	1.03±0.02	**75.5**	1.45±0.02	1.12±0.02	**77.3**
Schist B	1.49±0.02	1.20±0.03	**80.9**	1.23±0.02	1.02±0.02	**83.5**
Performance Criteria -				Not available		
Acceptable			>7-			
Marginal			55-7-			
Poor			<50			

TABLE 8 Re-evaluation of Initial and Net Adsorption Data for Bitumen 2

Net Adsorption according to SHRP		Re-evaluated adsorption according to Woodside et al[6]	
Aggregate	Percent	Initial Percent	Net (Percent)
Granite	79.4	45.7	37.1
Gritstone	79.8	37.1	30.5
Schist A	77.3	51.7	41.2
Schist B	83.5	43.9	37.9
Performance Criteria -		Not available	
Acceptable	>70		
Marginal	55-70		
Poor	<50		

TABLE 9 Re-evaluated and SHRP Results for Irish Aggregates and Emulsions

AGGREGATE	Adsorption				
	Calculated according to SHRP			Calculated according to Woodside et al[6]	
	A_i Initial Adsorption (mg/g)	A_n Net Adsorption (mg/g)	% NA	Initial (percent)	Net (percent)
		EMULSION 1			
Basalt	1.54±0.06	1.02±0.04	66.5	55.0	37.9
Granite	1.09±0.08	0.70±0.03	63.8	38.9	25.7
Gritstone	1.28±0.03	1.05±0.04	82.0	45.7	38.6
Limestone	1.45±0.05	1.30±0.07	90.1	51.8	47.8
Sandstone	1.35±0.07	1.18±0.03	87.9	48.2	43.4
Schist A	1.35±0.04	1.03±0.03	75.9	48.2	37.9
Schist B	1.27±0.03	1.09±0.07	85.8	45.5	40.1
		EMULSION 2			
Basalt	1.42±0.05	1.06±0.03	74.4	50.7	39.0
Granite	1.13±0.05	0.72±0.04	64.3	40.5	26.8
Gritstone	1.35±0.07	0.96±0.06	71.3	48.2	35.3
Limestone	1.28±0.06	1.16±0.05	90.6	45.7	42.7
Sandstone	1.46±0.02	1.18±0.03	81.0	52.1	43.4
Schist A	1.46±0.07	1.04±0.05	71.3	52.1	38.2
Schist B	1.20±0.02	0.97±0.03	80.8	42.9	35.7
		EMULSION 3			
Basalt	1.37±0.03	1.06±0.04	77.4	48.9	39.0
Granite	1.04±0.02	0.84±0.03	80.5	37.1	30.9
Gritstone	1.17±0.03	0.93±0.03	79.5	41.8	34.2
Limestone	1.49±0.05	1.15±0.05	77.2	53.2	42.3
Sandstone	1.27±0.02	0.89±0.03	70.4	45.4	33.1
Schist A	1.22±0.03	1.04±0.01	85.2	43.6	38.2
Schist B	1.39±0.03	1.04±0.02	75.1	49.6	38.2
Performance Criteria -				Not available	
Acceptable			>70		
Marginal			55-70		
Poor			<55		

FIGURE 1 Adsorption of a bitumen onto four different aggregates.

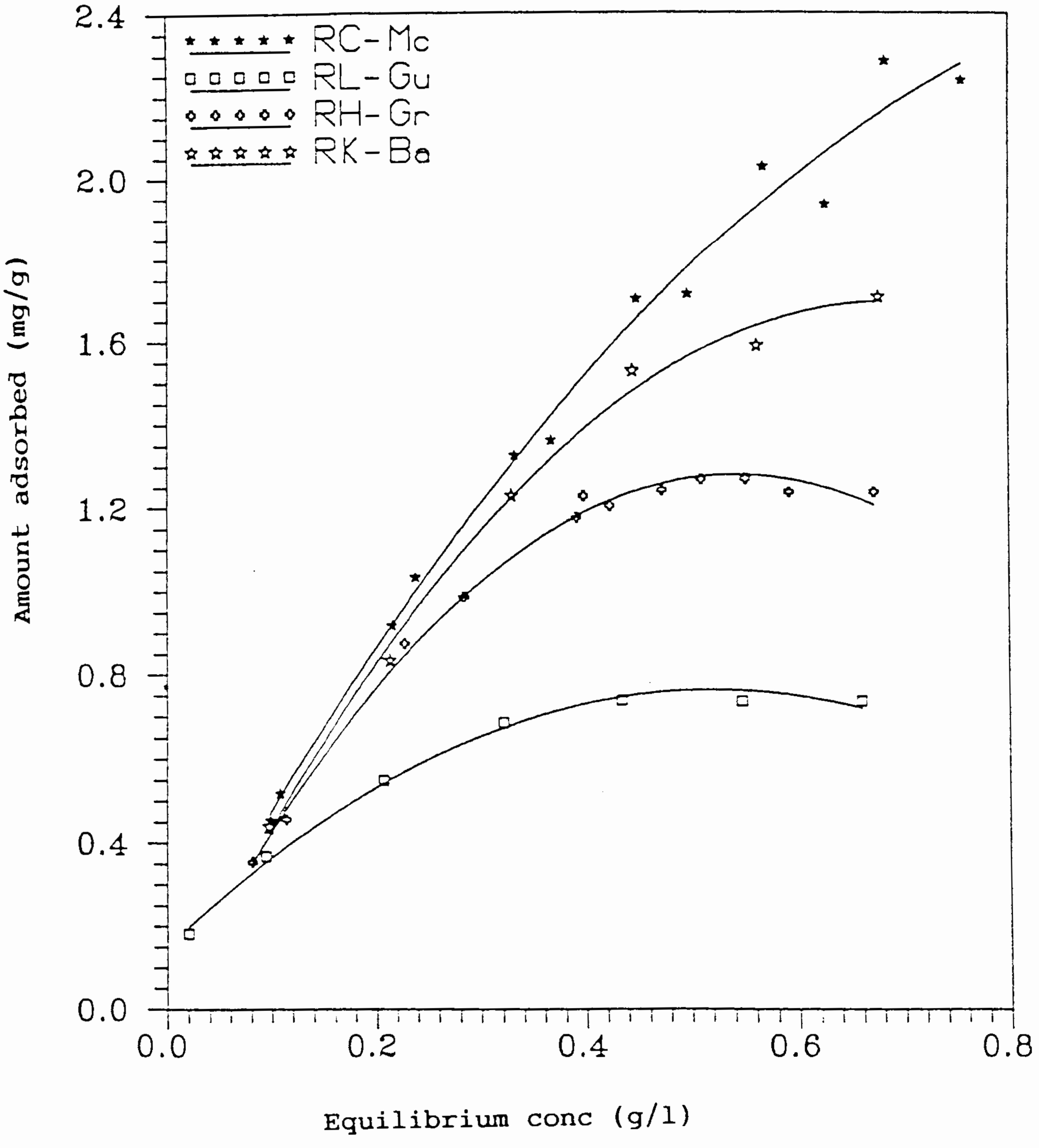
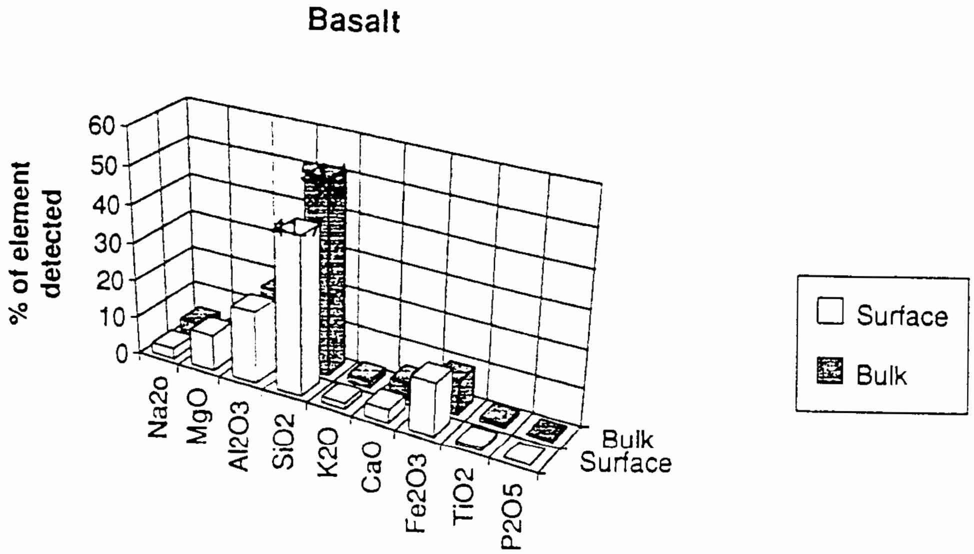
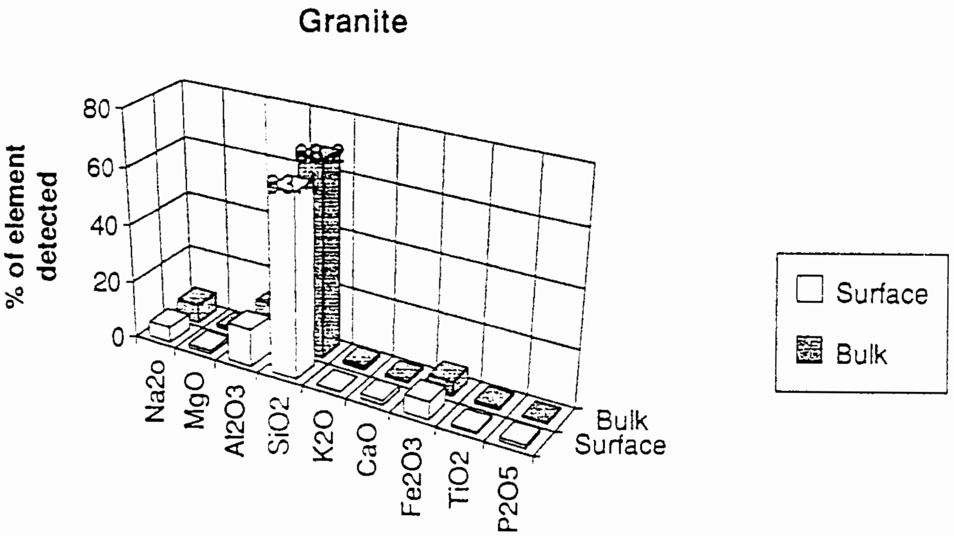
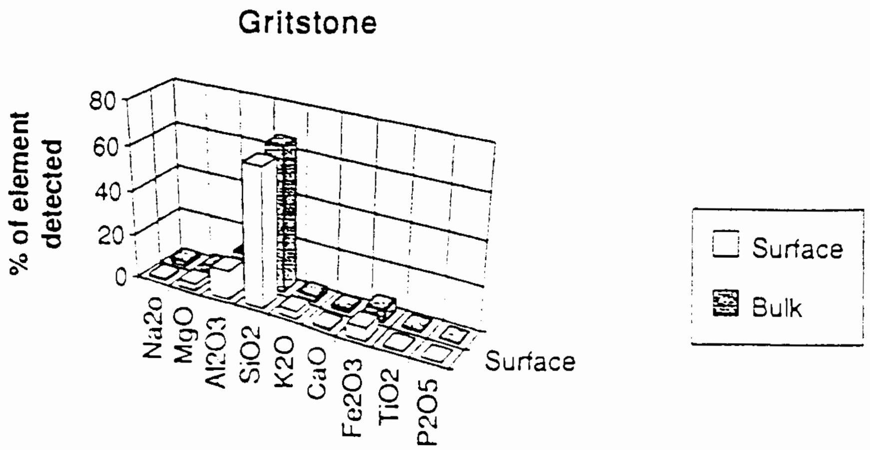


FIGURE 2 Composition: Chipping surfaces and crushed dust.







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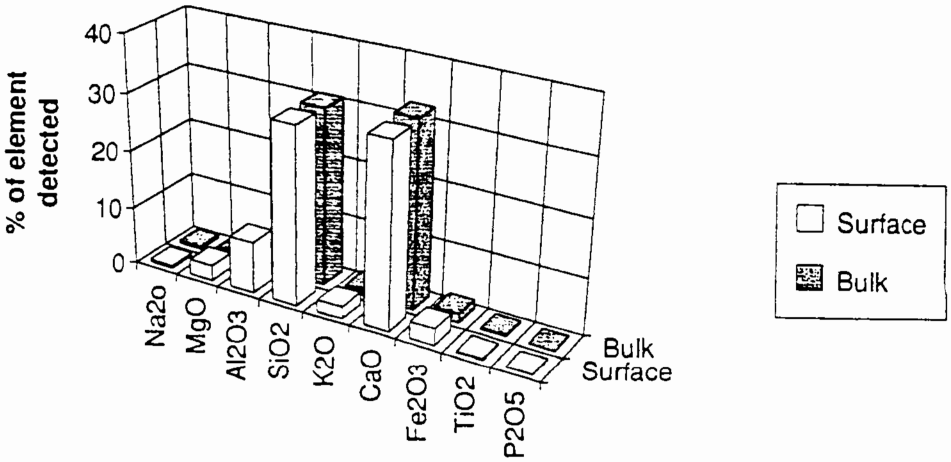

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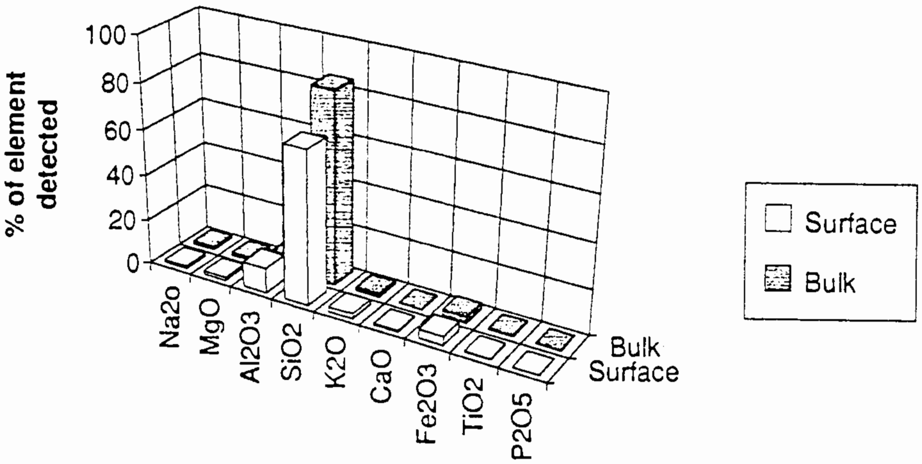
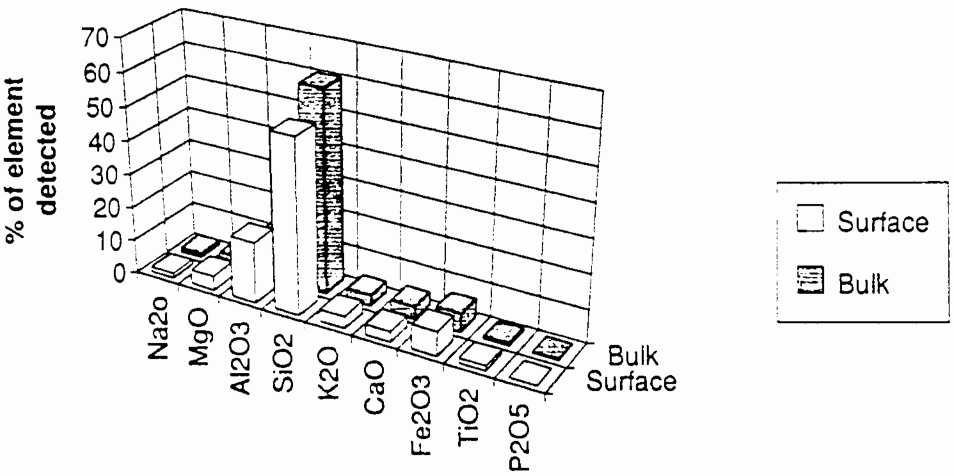


Schist A



Schist B

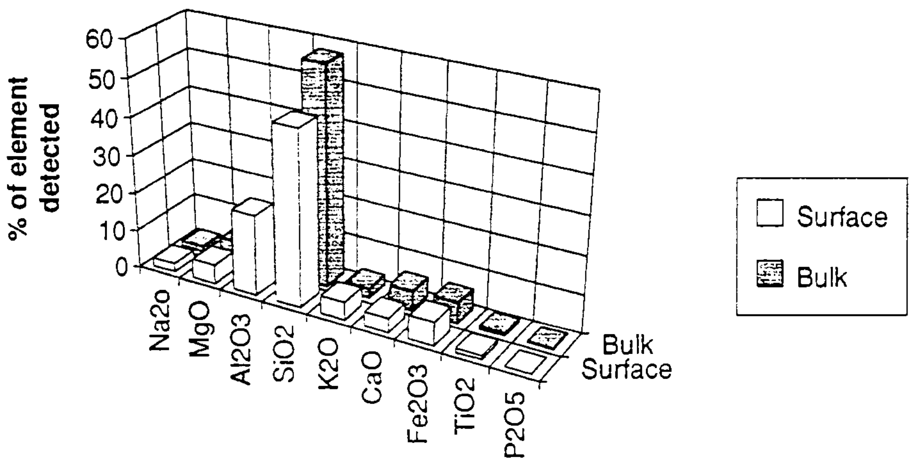

% of element detected
60
50
40
30
20
10
0
Na2o
MgO
Al2O3
SiO2
K2O
CaO
Fe2O3
TiO2
P2O5
Bulk
Surface
Surface
Bulk

FIGURE 3 NAT results: Irish aggregates and bitumens

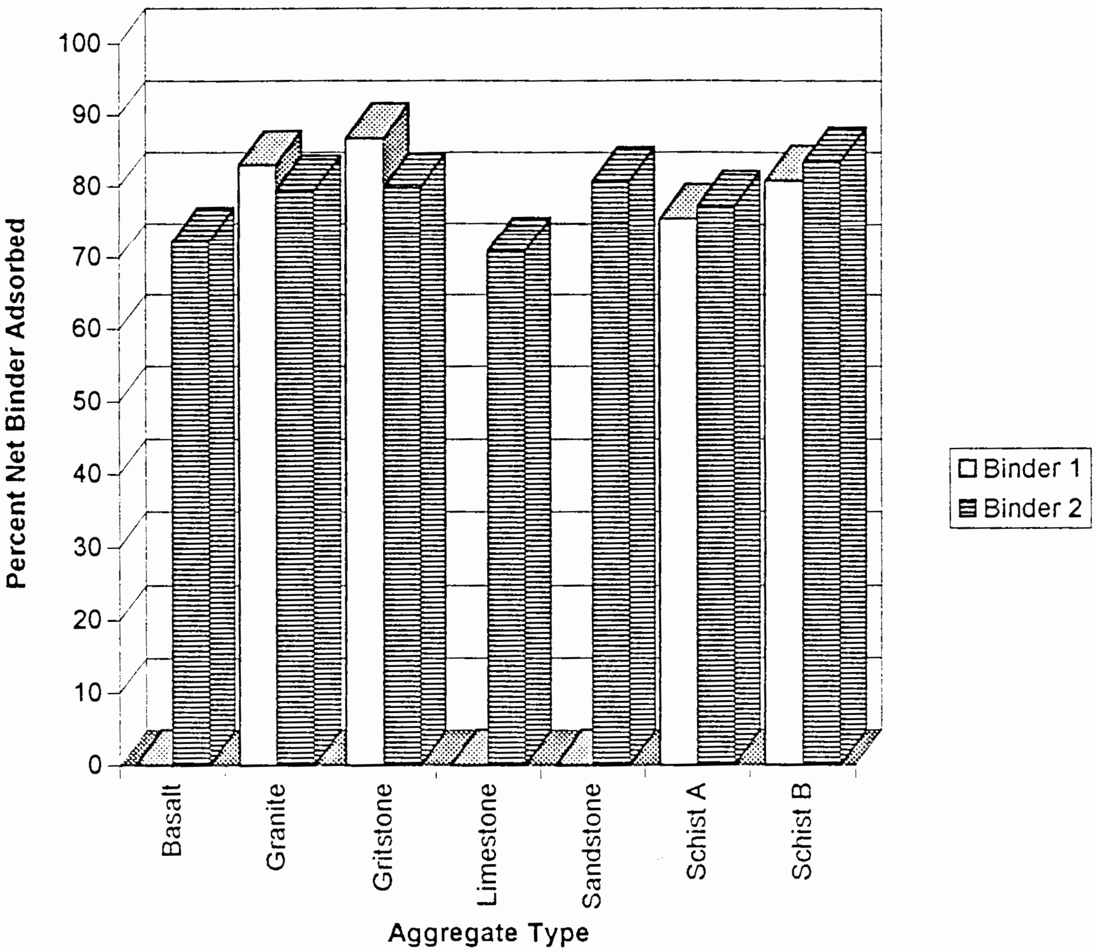


FIGURE 4 Bitumen/Aggregate interaction diagram

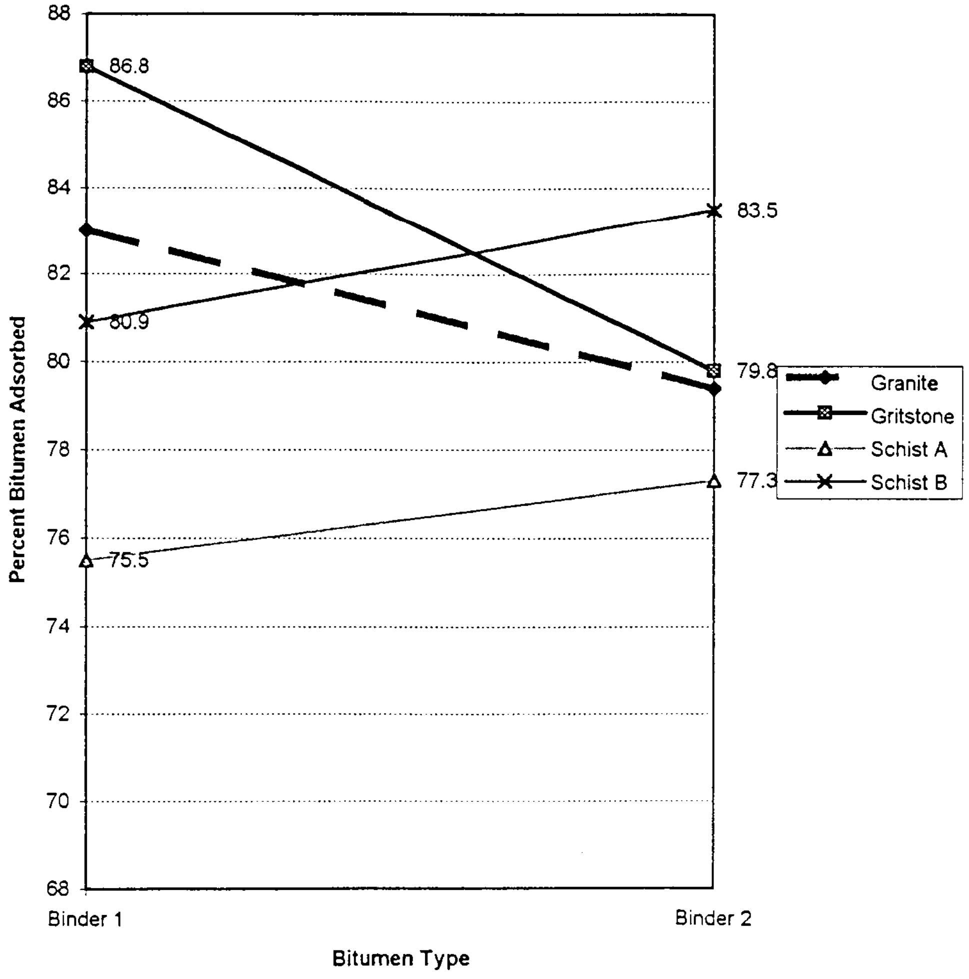


FIGURE 5 NAT results: Irish aggregates and bitumen emulsions

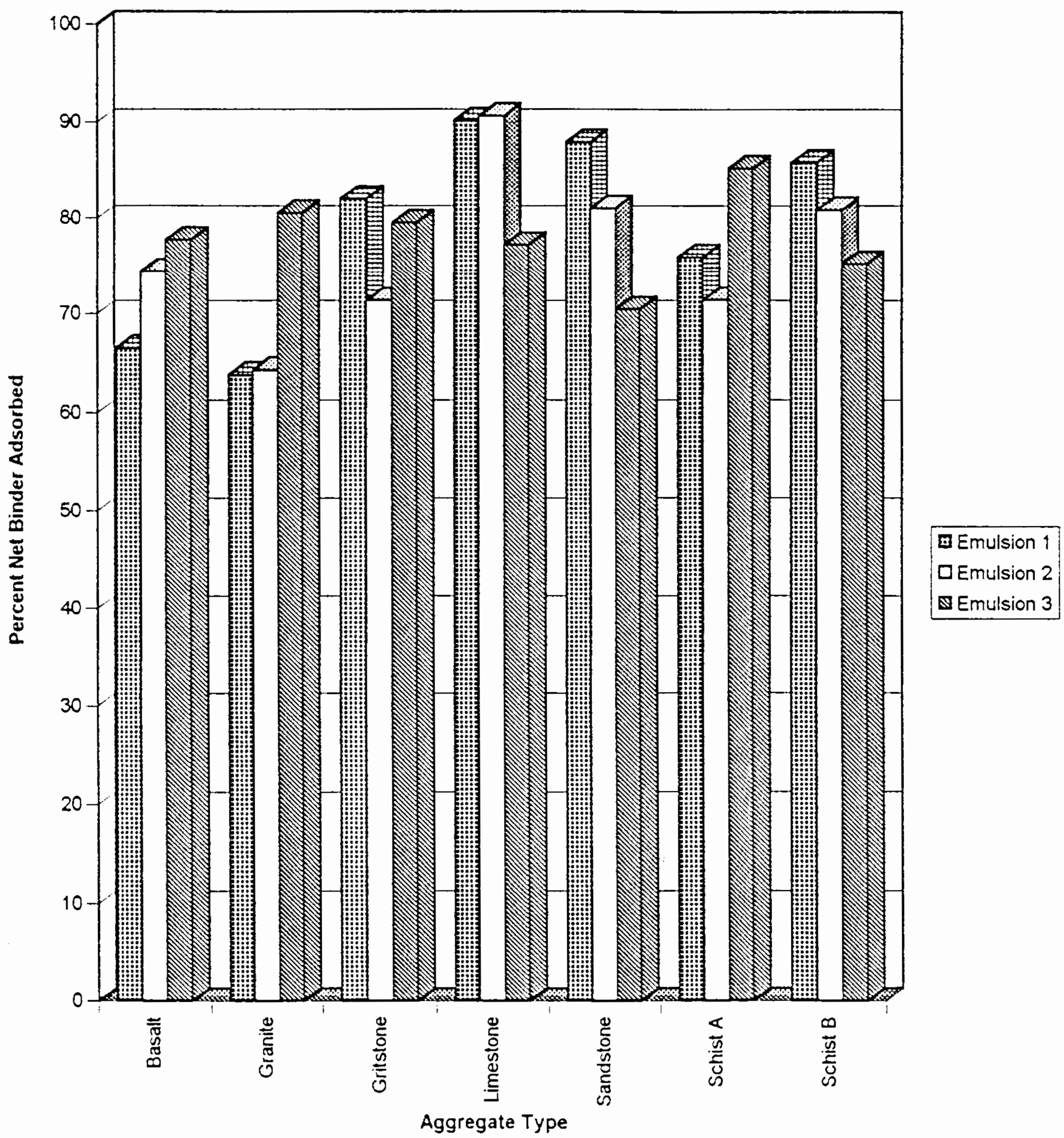
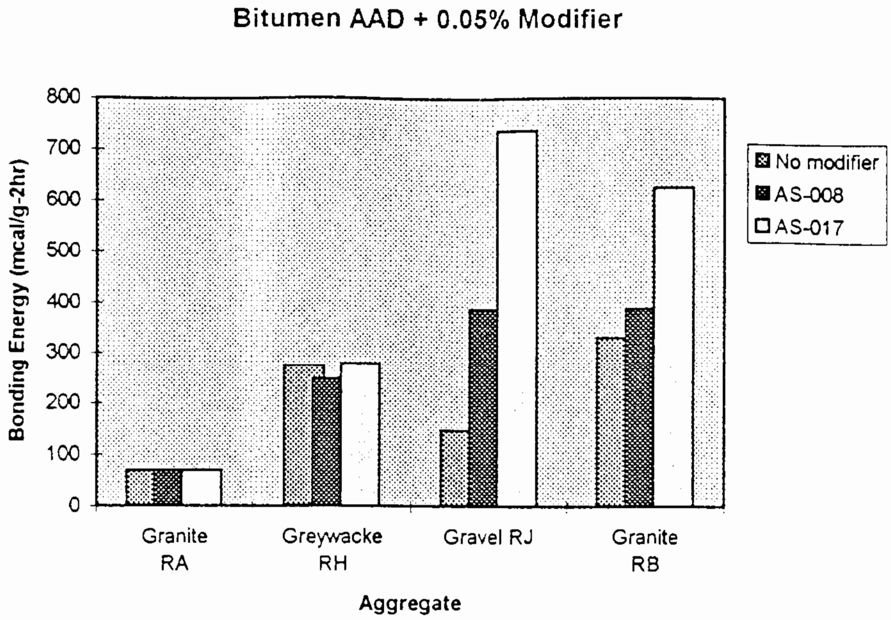


FIGURE 6 NAT results: Influence of anti-strip agents



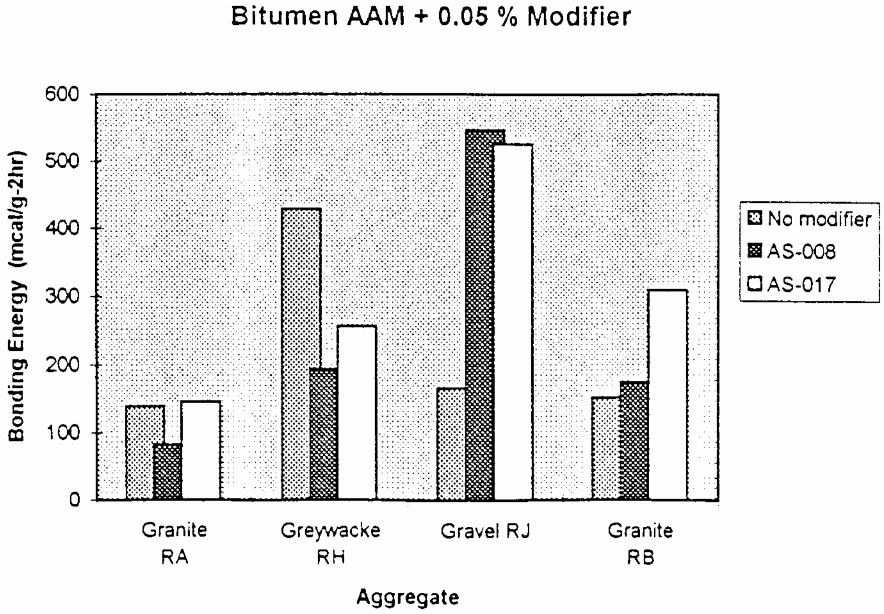


FIGURE 7 NAT results: Initial vs net adsorption. Calculated by Woodside method