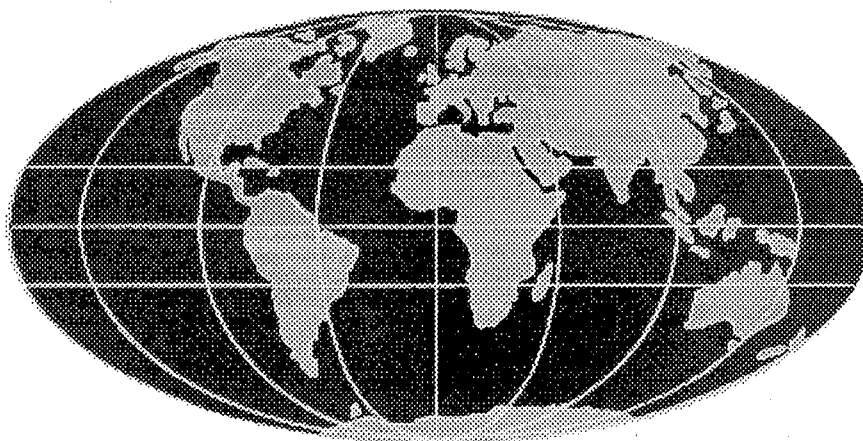


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PA1285/1992

OBIKA, B, R J FREER-HEWISH and D NEWILL, 1992. Physico-chemical aspects of soluble salt damage to thin bituminous road surfacing. In: *Pre-Conference Proceedings of International Conference on the Implications of Ground Chemistry and Microbiology for Construction, University of Bristol, 29 June-1 July 1992.*

PHYSICO-CHEMICAL ASPECTS OF SOLUBLE SALT DAMAGE TO THIN BITUMINOUS ROAD SURFACING

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ABSTRACT

Soluble salt contamination of highway construction materials occurs in climatic zones where evaporation exceeds precipitation. This results in an upward migration of moisture to the surface, where salts present in solution are precipitated. These climatic zones cover large areas of Australia, Africa, The Middle East and North and South America.

Existing guidelines and recommendations for road design and construction in saline areas are based mainly on experience of local materials and conditions without a full understanding of the damage mechanism. This has resulted in conflicting advice on how to prevent salt damage.

A laboratory simulation approach to understand the salt damage process has been reported. This paper is concerned with an appreciation of the geochemical processes, which are important for current laboratory and field testing programmes.

INTRODUCTION

Damage to bituminous surfaced roads and runways in arid, semi arid and warm coastal environments has, in certain cases, been attributed to the action of soluble salts. The existing literature on the subject does not explain the fundamental mechanisms, however, and this has led to varying advice on how to prevent salt damage. There is a need to appraise the

existing recommendations for the design and construction of roads and runways in saline environments, based on a better understanding of the salt damage process.

Soluble salt damage to bituminous surfacings is caused by upward migration of salts under an evaporation gradient. At or near the surface the salts in solution become supersaturated and crystallise. This can cause a physical degradation of the bituminous surfacing, often in the form of blistering, doming or general powdering/disintegration (Plate 1). Bituminous prime coats and surface dressings are particularly susceptible to salt damage.

Salt damage will only occur in climatic zones where evaporation exceeds precipitation. Under such conditions, moisture is drawn to the surface where soluble salts present in solution are precipitated. These climatic zones cover large areas of Australia, Africa, The Middle East and North and South America. A detailed review of the extent of occurrence of salt damage and existing recommendations in these areas is given in Obika, Freer-Hewish and Fookes, 1989.

The work reported in this paper is part of an ongoing study at the University of Birmingham initially sponsored by the British Overseas Development Administration (ODA) following the occurrence of salt damage to the bituminous surfacing of a runway in the West Indies. The work has included extensive laboratory climatic cabinet simulation and field studies in the West Indies and Chile. Experimental road sections have also been constructed in Botswana under a two year research programme sponsored by Overseas Unit, Transport and Road Research Laboratory. The experimental road sections are currently being monitored and will be reported in a later paper.

This paper describes the pertinent aspects of salt solubility, movement, crystallisation and crystal pressures which can lead to a better understanding of the salt damage mechanism.

EXISTING RECOMMENDATIONS

The few published papers on salt damage deal with local environments and materials and this has resulted in a variety of recommendations for damage prevention which are often conflicting. Early published reports in Australia (Cole and Lewis 1960) and in South Africa (Weinert and Clauss 1967) suggested upper limits of sodium chloride (NaCl) content and sulphate content (as SO_3) of 0.2% and 0.05% respectively. Subsequent work (Netterberg et al 1974; Fookes and French 1977, Januszke and Booth 1984, Obika et al 1989) have shown that these limits are probably not applicable universally. In addition to imposing maximum salt limits the recommendations for salt damage prevention also include: rolling the bituminous surface, preferred use of bituminous emulsion binders rather than bituminous cutbacks for prime coats and minimising time duration between base construction and surfacing. The relative effectiveness of these measures is discussed in Obika et al (1989).

Horta (1985) provided the first detailed examination of the damage mechanisms and suggested possible approaches to developing preventative measures. He described the occurrence of salt damage in various parts of North Africa and noted the presence of NaCl filamentous crystals in many damaged surfacings. This work also drew attention to some critical crystal growth factors and the need for a rational approach to understanding the damage mechanism.

SALT DAMAGE MECHANISMS AND CRYSTAL PRESSURES

Clearly, the development of methods of prevention and repair of salt damaged pavements should be based on consideration of the fundamental damage mechanism. Evaporation, salt migration, solubility, crystallisation and crystal pressures are discussed below.

EVAPORATION AND MIGRATION OF DELETERIOUS SALTS

Salt migration occurs by capillary movement of saline solution due primarily to evaporation. The effect of evaporation is to provide a suction

gradient which is greater than the opposing gravitational gradient encouraging the movement of moisture towards the pavement surface.

There are two stages in the drying process (Ward 1975). The first is characterised by a constant rate of evaporation governed largely by climatic and pavement surface conditions and the second by a declining rate of evaporation governed mainly by the ability of the pavement layers to transmit moisture to the evaporating surface. Thus at first the suction gradient increases as the pavement becomes drier, but at the same time the moisture films through which water movement occurs become thinner and fewer in number. The rate of moisture movement therefore, decreases as the pavement dries. This reduced moisture movement and reduced evaporation is further encouraged by the decreasing moisture gradient. In addition, if salt crystals are precipitated at the surface they will retain moisture at the pavement surface and reduce the moisture gradient further.

Thus the most rapid increase in salt content at the pavement surface may occur soon after pavement construction, followed by a second stage of declining rate. The time required to reach this second stage will depend on the initial salt content of the material, the moisture condition of the pavement and the climatic conditions.

A typical relationship between salt content at the unsealed pavement surface and time is shown in Figure 1. This has been developed from laboratory climatic cabinet simulations of a West Indian climate using crushed West Indian oolitic limestone. As noted above, the relationship may vary but it is important to recognise that rapid upward salt migration can occur within 48 hours after construction or ground excavation. Design parameters based on pre-construction or initial salt content determinations may not always be appropriate.

SOLUBILITY AND CRYSTALLISATION OF DELETERIOUS SALTS

Only those salts which are soluble in water can migrate to the surface of the pavement. The solubility of a given salt is related to its crystallisation thresholds. The solubility of sodium chloride increases only slightly with

increase in temperature, whereas sodium sulphate, magnesium sulphate and sodium carbonate show a rapid initial increase with temperature. At typical ambient temperatures of 30°C, common in hot climates, the solubility of the salts is near its maximum.

Salt crystallisation evolves through three stages:

- attainment of supersaturation;
- formation of crystal nuclei;
- growth of crystals.

When a solution contains more dissolved salts than the equilibrium concentration it is said to be supersaturated. Supersaturation is the most important variable influencing the magnitude of disruptive pressures for a given crystal. It can be induced in a number of ways including cooling, evaporation of solvent and reduction of the solubility of one salt by another.

SALT CRYSTAL TRANSITION POINTS

Certain salts, such as sodium sulphate and sodium carbonate, have hydrates whilst others such as sodium chloride do not. A hydrate is a salt which contains water of crystallisation. Hydration can occur when the temperature is at or below the transition point of a given salt. At the transition point there is a reversible change from an anhydrous salt to a hydrate or from a lower to a higher hydrate. For a given temperature of hydration, at or below the transition point, there is also a critical ambient relative humidity which must be exceeded if hydration is to take place.

In the presence of excess moisture (r.h. >60%) sodium sulphate (Na_2SO_4) forms a decahydrate ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) at 32°C with an associated volume change in excess of 300% (Sperling and Cooke 1985). Sodium carbonate (Na_2CO_3) changes to its heptahydrate state ($\text{Na}_2\text{CO}_3 \cdot 7\text{H}_2\text{O}$) at 31°C with similar crystal volume changes. However, at very low humidities, such as 30%, hydration will not normally occur.

Sodium chloride does not have a hydrate but tends to absorb moisture from the atmosphere and dissolve at relative humidities above 76%. Below

this humidity it recrystallises with the development of associated crystallisation pressures. This is discussed below.

In many hot dry lands these crystal thresholds are crossed at least once in a single day creating pressures sufficient to disrupt pavement surfacings.

Salt Mixtures

The relative ability of a given salt to cause damage depends to a large extent on its solubility and crystallisation properties. However, both the solubility and transition point of a given salt can vary depending on what other salts are present. For example the transition point of sodium sulphate decahydrate is reduced from 32°C to below 20°C in the presence of a low concentration of sodium chloride. For practical purposes the design approach for prevention of salt damage would be normally based on the dominant salt but an awareness of the above is essential.

Sodium Chloride Threshold

The presence of sodium chloride crystals, known as halite, has been reported in several cases of salt damage. It is also the most widely occurring natural salt and requires particular attention.

Increasing dissolved salt concentration decreases the chemical activity of H_2O in a solution and as a result lowers the equilibrium water vapour pressure. This arises because when salts are added to water, some fraction of the water molecules are structurally bound to the salt ions, reducing the chemical activity of H_2O . The extent of lowering of the equilibrium water vapour pressure depends on the particular type of dissolved salt present. For NaCl solutions, the vapour pressure lowering is approximately 23% at normal ambient temperature (Kinsman 1926). Thus in a concentrated NaCl solution such as a brine, the chemical activity of H_2O is 0.76 (the chemical activity of H_2O in pure water is unity) and the equilibrium vapour is achieved at a relative humidity of 76% as opposed to 100% vapour pressure. Thus for the NaCl solution to be evaporated, relative humidities less than 76% must prevail in the overlying atmosphere.

It follows, therefore that NaCl crystals, can only be precipitated and accumulated in those areas where the mean relative humidity of the atmosphere is less than 76%. Mean relative humidities of many low latitude coastal and arid regions, commonly fluctuate between 70 and 80% (Kinsman 1926). At all relative humidities above 76%, there will be a net flux of water vapour from the atmosphere into the salt solution. At all relative humidities below 76% evaporation of the solution and precipitation of halite will occur.

Halite may crystallise and redissolve at least once in a single day creating high heaving pressures at the pavement surface.

In the sabkhas of Abu Dhabi, Trucial Coasts and Persian Gulf, unsealed roads which are dry at mid-day, frequently become wet at dawn as a result of moisture attracted by halites present on the unsealed road surface.

It is concluded that in climatic zones where the relative humidity remains high (above 76%) all year round, salt damage due to halites is unlikely as crystals will not precipitate from capillary solution in substantial quantities. Where the daily fluctuations of relative humidity cross the 76% threshold, damage due to halites should be anticipated.

Filamentous Crystals

The occurrence of filamentous crystals (whiskers) in cases of salt damaged roads and runways has been documented (Horta 1985, Cole and Lewis 1960). Detailed studies of damaged field and laboratory surfacings using a scanning electron microscope (Obika and Freer-Hewish 1988) have shown that whiskers of sodium-chloride salts were responsible for the damage to bituminous surfacings.

Whiskers, often referred to as filamentous or fibrous are high super-saturation, elongated crystals with higher than normal strength and crystallisation pressures. There are several known properties and features of this type of crystal which are fundamental in understanding the mechanism and prevention of salt damage. For example it has been shown

that sodium chloride(NaCl) whiskers will grow preferentially in substrates of finer porosity. Thus base courses with high fines content are particularly susceptible to salt heave. Horta (1985) noted that the Old Adrar Airport in Algeria with a base course high in clay content , showed early heave caused by NaCl whisker growths. It is known that the growth of whiskers can be inhibited by the presence of low supersaturation crystals of the same chemical composition or by impurities (Nabarro and Jackson 1958); for example sodium chloride whiskers will not normally grow from solutions when sodium chloride cubic crystals are also present.

SALT CRYSTAL PRESSURES

Opinions differ on the relative importance of stresses produced during salt crystallisation, growth, hydration and thermal expansion on the breakdown of materials. Evans (1970) has compiled a review of salt crystallisation relevant to salt weathering of rocks and lists the various arguments concerning the origin and nature of salt crystal pressures. Theoretical and observed pressures indicate crystal growth and hydration may be important(Correns 1949, Wellman and Wilson 1965, Winkler 1975).

Crystal Growth Pressures

The pressure (P) exerted by crystal growth can be expressed as (Correns 1949):

$$P = \frac{RT}{V_s} \text{Log } c/c_s \quad [1]$$

where R is the gas constant of the ideal gas law, T is the temperature in degrees Kelvin, V_s is the volume of the solid salt, c is the actual concentration of solute during crystallisation and c_s is the concentration of the solute at saturation.

The major factor influencing the magnitude of crystal growth pressure is the level of supersaturation c/c_s. Pressures in excess of 500 atmospheres can develop at low supersaturation (Obika and Freer-Hewish 1988).

Hydration Pressures

The hydration pressure (P_h) is given by (Winkler 1975) :

$$P_h = \frac{(nRT)}{V_h - V_a} \times 2.3 \log \frac{P_w}{P_{w1}} \quad [2]$$

where n is the number of moles of water gained during hydration, V_h is the volume of hydrates in cubic centimetre per gram-mole, V_a is the volume of the original salt before hydration in cubic centimetre per gram-mole, P_w is the vapour pressure of water in millimetres of mercury at a given temperature and P_{w1} is the vapour pressure of hydrated salt in similar units to P_w .

The rate of hydration is also important in determining the overall destructive effect of a particular type of salt. The hydration of sodium sulphate heptahydrate to the decahydrate may repeat several times in a single day (Wellman and Wilson 1965). The hydration takes about 20 minutes. The ability of magnesium sulphate to cause more damage than sodium sulphate in laboratory testing is attributed to the faster hydration rate of magnesium sulphate.

Thermal Expansion Pressures

Cooke and Smalley (1968) suggested that thermal expansion of salts within rock pores may create pressures sufficient to cause rock weathering. These pressures arise because of the lower thermal coefficient of expansion of rock minerals when compared with that of certain salt crystals. The importance of pressures due to the above mechanism has not been investigated in any detail and further work is required.

Very few measurements of crystal growth pressures have been recorded. The earliest known observations are those of Becker and Day (Evans 1970). The authors noted that alum and CaSO_3 crystals growing between two plates of glass can lift one kilogram (kg) through several tenths of a millimetre. Taber (1916) observed that growth of loaded crystals was slower than unloaded crystals. However, the growth of the former could be

increased by raising the level of supersaturation. Mosebach (1951) measured pressures in excess of 47 atmospheres by growing copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) crystals. The author also demonstrated that different crystal faces have different abilities to grow against pressure. For example, the (010) face of potassium chromate ($\text{K}_2\text{Cr}_2\text{O}_7$) was found to grow against a pressure twice the limit for the (001) face. Correns (1949) also found that certain faces of the alum crystal did not show any growth whereas other faces grew against pressures up to 42 atmospheres.

MECHANICS OF CRYSTALLISATION AT BITUMEN - BASE INTERFACES

Correns (1949) compared observed and calculated pressures using equation 1 and found significant variations at high supersaturation which he explained in terms of the phase boundary tensions. Thus if the phase boundary tension between crystal (a) and solution (b) is σ_{ab} , that between the substance of the plates (c) (under and above the crystal) and the solution (b) is σ_{bc} and that between crystal (a) and substance (c) is σ_{ac} then the following relation must hold for the crystal to grow.

$$\sigma_{ac} > \sigma_{bc} + \sigma_{ab} \quad [3]$$

Correns found that although the crystal grew against pressures when sandwiched between two glasses, the same crystal (with identical face orientation) did not grow when sandwiched between two mica plates. In the latter case the author assumed $\sigma_{ac} = \sigma_{bc} + \sigma_{ab}$. The surface tension theory may be extended to the case of salt crystallisation at the bitumen-base interface of a road pavement, see Figure 2. If the bitumen and the soil form the upper and lower plates respectively, the phase boundary tensions can be expressed as follows:

Between :	bitumen (b) and crystal (c)	σ_{bc}
	bitumen and solution (s)	σ_{bs}
	crystal and solution	σ_{cs}
	crystal and soil (p)	σ_{cp}
	solution and soil	σ_{sp}

In order that the crystal might grow the following relation must prevail:

$$\sigma_{bc} + \sigma_{cp} > \sigma_{bs} + \sigma_{cs} + \sigma_{sp} \quad [4]$$

The above relationship demonstrates the importance of the chemical properties of the pavement materials. For example, any additive or inherent property of the bitumen or the soil which is likely to increase the phase boundary tension between the materials and the salt crystal will tend to increase the potential for the salt crystal to grow. Additives may also be used to reduce the boundary tensions involved, thereby reducing the potential for crystal growth.

INFLUENCE OF BASE POROSITY ON CRYSTALLISATION AT BITUM-BASE INTERFACES

For materials of equal strength, those with large pores separated from each other by microporous regions will be the most susceptible to salt weathering (Wellman and Wilson 1965).

The work required to be done during crystal growth on one face of a crystal is equal to $(P_1 - P_s)dV$. Where P_1 is the pressure in the solution, P_s is the solid, and dV is the increase in volume. This must equal the work done in extending the surface, σdA , where σ is the boundary surface tension between the crystal face and its saturated solution and dA is the increment of area.

Thus

$$P_1 - P_s = \sigma \frac{dA}{dV}$$

Consider salt crystals growing at the bitumen-base interface where the micropores formed by the interface are larger than those in the base. The crystals will grow first at the interface as a result of evaporation. The growth continues until the interface pores are filled. As $P_1 - P_s = \sigma dA/dV$, for the crystals to extend by growth into the smaller pores of the base would involve a high increase in area (dA) relative to volume (dV). This would require an unduly large amount of work to be done. The crystal will therefore continue to grow at the bitumen-base interface, until the pressure is sufficient to heave or rupture the bituminous surfacing. The above

mechanism indicates that heaving pressures will be greater in a base with high fines content and this is consistent with field observations.

The mechanism whereby salt crystals grow at the bitumen-base interface rather than the top (exposed side) of the bitumen requires some consideration. A possible explanation is that the bituminous layer may act as a semi permeable membrane. Upon reaching the bitumen-base interface, moisture only is allowed to pass through the microporous bitumen generally in the vapour phase.

An alternative explanation, however, may be that nucleation is favoured at the interface due to the existence of crystals accumulated on the base prior to bitumen application. These crystals continue to grow until the interface pores are filled. As a large chemical potential would be required for the crystals to extend into the small micropores of the bitumen, the crystals continue to grow at the bitumen-base interface causing an upward heave of the bituminous layer.

Crystallisation under applied load

By considering the relation between pressure solution and the force of crystallisation, Weyl (1959) developed an expression for the pressures involved during crystallisation. Thus, if the level of supersaturation and the stress coefficient of solubility are known, the pressure against which a given crystal will grow can be determined.

The author reasoned that the phenomenon of pressure solution and the force of crystallisation is the result of removal or deposition of mineral matter in the region of contact between two mineral grains. The force of crystallisation is therefore the antithesis of pressure solution. In developing the theory, it is assumed that a film of solution exists, a few atoms thick, between two crystal grains through which diffusion occurs. Evidence for the existence of this film is given by Evans 1970, Correns 1949 and Weyl 1959.

The rate of diffusion through this film depends on the grain size, the effective normal stress (total normal stress minus hydrostatic pressure) between

the grains, the diffusion constant in the solution film, the film thickness and the stress coefficient of solubility; the latter being related to the tendency of crystals to dissolve under stress.

Weyl demonstrated that for a given set of parameters, as the average effective stress is increased, the force of crystallisation occurs by growth with a hollow centre. Further increases in effective stress decreases the rate of crystal growth as the radius of the hollow centre decreases until it disappears. As the effective stress is further increased, the rate of crystal growth continues to decrease until the stress across the film is constant and there is, therefore, no diffusion gradient. A further increase in effective stress changes the direction of solute diffusion and pressure solution occurs.

The force against which crystallisation can occur increases with the degree of supersaturation and with decreasing stress coefficient of solubility. Weyl gives a practical example; calcite has a fractional change of solubility with hydrostatic pressure of approximately 10^{-3} per atmosphere, therefore at 1% supersaturation, calcite can be expected to crystallise against a force of 10 atmospheres. If supersaturation is higher it will crystallise against a proportionately higher force.

THE EFFECT OF CLAY ON CRYSTALLATION PRESSURES

If a clay film is present within the solution film bordering the two crystal grains discussed above, the effect will be to increase the force of crystallisation. The clay film consists of a series of platelets with associated water films. These water films provide significantly increased rates of solute diffusion which result in increased rate of crystallisation.

THE HALITE DAMAGE PROCESS

The foregoing may be related to the halite damage process. The damage process is initiated by the accumulation of cubic halite crystals on the surface of the base before the bituminous surfacing is applied. Under these conditions evaporation is moderate but continuous. The capillary solution

reaching the surface is never allowed to reach the high levels of supersaturation as cubic halite crystals nucleate and crystallise out instantaneously on reaching the surface. The cubic crystals have comparatively low surface free energy, exert lower pressures and form at lower levels of supersaturation. Cubic crystals are the stable form of halites.

Although when the bituminous surfacing is applied the evaporation rate is immediately reduced, a moisture gradient still exists through the pavement and further migration of saline moisture to the surface occurs. The moisture is unable to escape rapidly and the salt crust on top of the base maybe partially redissolved. The resulting salt solution is now at a higher level of supersaturation. The high supersaturation combined with the confined volume between the base and surfacing provide conditions favourable to filamentous crystal growth. The filamentous crystals (or whiskers) have high surface free energy and exert higher crystal pressures. Evaporation still occurs but at a much slower rate through the bituminous surfacing, aided by drying shrinkage cracks. The filamentous crystals nucleate and grow perpendicular to the surface of the base between the bitumen and the base. As it is energetically less feasible (Wellman and Wilson 1965) for the crystal to extend into the micropores of the base or bitumen, it continues to grow at the interface, lifting the bituminous surfacing by exerting pressures along the crystal longitudinal axis.

Further growth of filamentous crystals takes place slowly as more salt migrates to the surface. Where the surfacing cracks, increased growth occurs as evaporation accelerates, drawing more salt to the surface. Early trafficking is often beneficial as this development of domes and blisters created by the salt crystals are suppressed by tyre pressures. This prevents increased evaporation and hence crystallisation.

CONCLUSION

Certain climatic conditions are essential for salt damage of bituminous surfacings to occur. Damage is most likely where there is a large diurnal change in temperature and relative humidity and where evaporation is generally high. In such environments repeated crossing of the crystal

formation thresholds may lead to the development of destructive pressures at the pavement surface. The pressures developed by salt crystallisation can be high and depend to a large extent on the level of supersaturation of the saline solution.

Where contaminated pavement material contains more than one type of salt the crystallisation characteristics are difficult to determine. Existing recommended salt limits require careful interpretation in such cases and a small scale laboratory or field simulation (Obika and Freer-Hewish 1988) maybe useful in predicting whether damage may occur.

An improved understanding of the heaving mechanism caused by salt crystallisation between bituminous surfacing layers and base layers is assisting in the development of guidelines for preventative measures to avoid salt damage for wide ranging conditions.

ACKNOWLEDGEMENTS

The study described in this paper was sponsored by the British Overseas Development Administration (ODA). The paper is also published by permission of the Director of Transport and Road Research Laboratory, UK. The authors are grateful for information provided by Wallace Evans and Partners and the facilities provided by Sir Owen Williams and Partners Geotechnical Limited.

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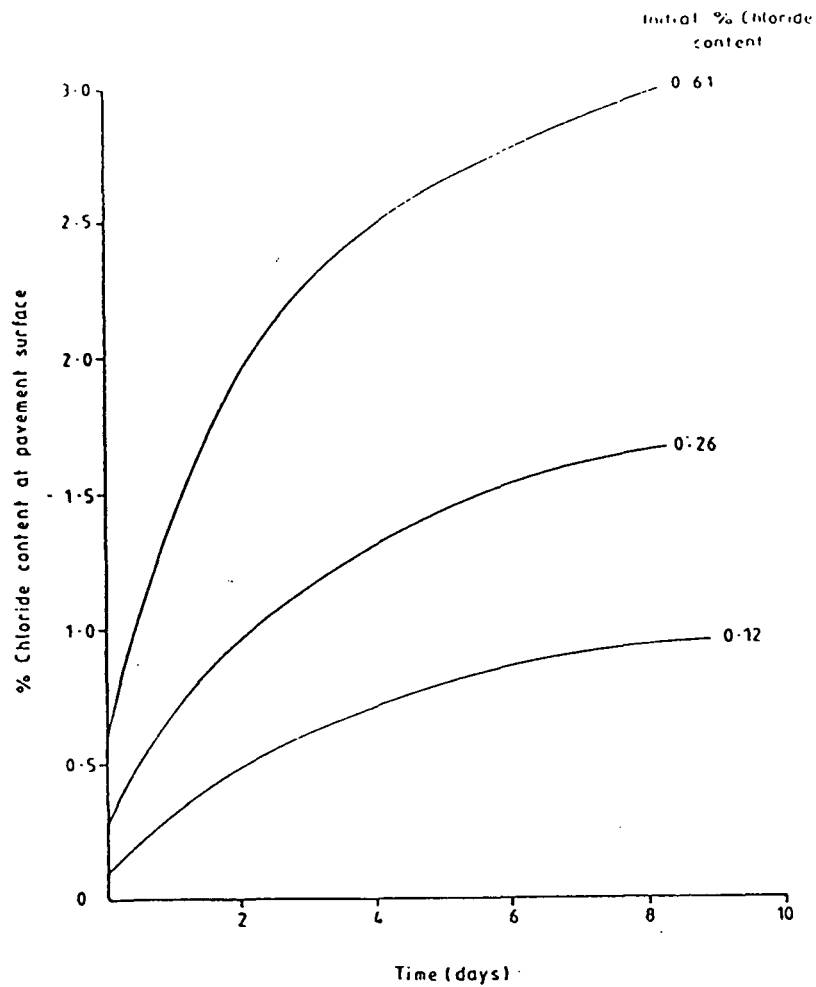


Figure 1: Relationship between initial chloride content and chloride content at pavement surface with time

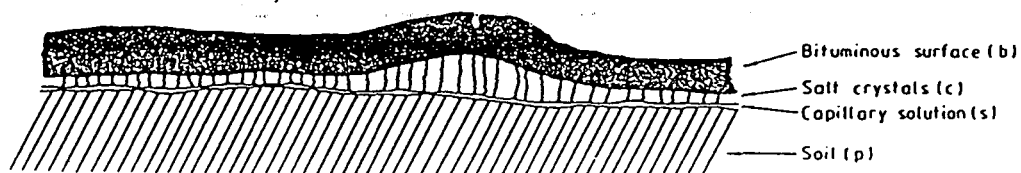
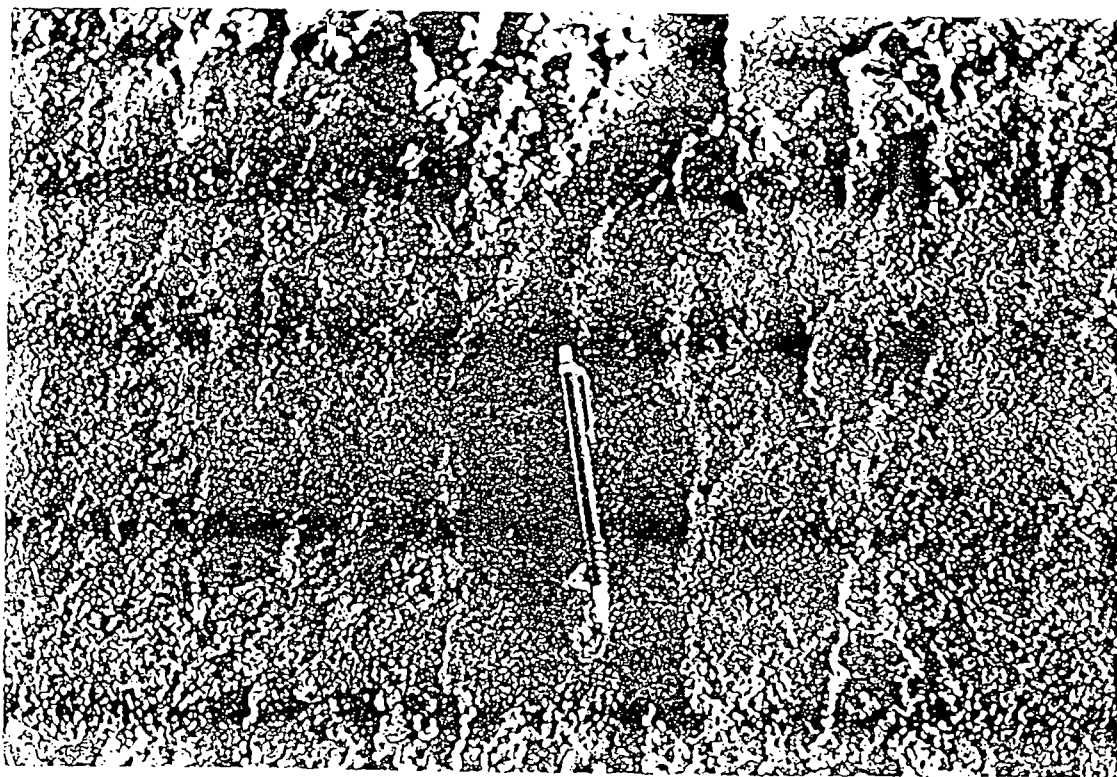


Figure 2: Salt crystallisation at bitumen-base interface



(a)



(b)

Plate 1 a) Doming of cape seal coat, Sua Pan Airstrip, Botswana
 b) Blistering of a bituminous cutback prime