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THE PERFORMANCE OF 2-NITROSO-1-NAPHTHOL CHELATING PIGMENT IN PAINT FORMULATION WITH GUM ARABIC AND POLYVINYL ACETATE AS BINDERS, PAPER I: UV-VISIBLE SPECTROSCOPY, VISCOSITY AND BREAKING STRESS OF THE PAINTS

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ABSTRACT:- Two binders, polyvinyl acetate (PVAc) to represent oil base paints and Gum Arabic for the emulsion types mixed with the necessary additives were combined with the pigment. Six different metallic salts (Zn^{2+} , Cu^{2+} , Fe^{2+} , Mn^{2+} , Ni^{2+} and Cr^{3+}) were then added in 100, 50, 33 and 25% mole equivalent concentrations based on the pigment concentration, to give 1:1, 1:2, 1:3, and 1:4 complex types respectively. It is reported here that the wavelength of maximum absorption () of the paint solutions in ethanol changed as the ratios of the salts were altered, however, the Tristimulus colour analysis provided more insight into these differences. The viscosities of the paint solutions increased generally as the salt concentration decreased. This effect was more pronounced with the Gum Arabic than with PVAc. The Gum Arabic binder recorded the highest breaking stress at 25% mole equivalent salt concentration in the case of Mn^{2+} , Cu^{2+} , Zn^{2+} and Cr^{3+} . The best breaking stress for Fe^{2+} and Ni^{2+} was at 50% mole ratio. With all the coatings from the Gum Arabic binder the 100% mole salt ratio recorded the least breaking stress. For the PVAc binder, Cr^{3+} , Zn^{2+} , and Mn^{2+} recorded their highest breaking stresses at 33% mole equivalent concentration. For Ni^{2+} and Cu^{2+} the highest breaking stresses were at 50% mole equivalent. However, the Fe^{2+} produced coating with increasing breaking stress as the salt concentration increased.

INTRODUCTION

Excellent description provided by Special Relativity (SR) of an elementary particle phenomenon is usually taken to imply that space-time is described by pseudo-Riemmanian geometry. This is because SR implies a Minkowski geometry for space-time, but the experimental existence of gravitational red-shift makes it impossible for the Minkowski geometry to apply globally. An obvious way to mesh the Minkowski geometries at various points is to have global Riemmanian geometry which the Minkowski geometry is tangent to at each space-time event. This is the situation in General Relativity (GR) which is a one-geometry description of physics. However, several attempts have been made to provide a two geometry description which includes inter-alia, Brans-Dicke theory, variable mass theory and string theories, resulting into

two conformally related geometries in which one geometry describes gravitational physics while in the other geometry matter plays out its dynamics. These two geometry theories violate the strong equivalence principle but preserve the weak equivalence principle. However, because both conformal and affine geometries lack the physically important notion of distance, the only most natural generalization of the Riemannian geometry describing the physical arena in which gravitational as well as non-gravitational physics take place is the Finsler geometry [1,2,3,4]. Now it has been logically demonstrated, at least at theoretical level, beginning with the work of Adler [5] and in subsequent works [6,7,8,9] that photon propagation in a background electromagnetic field and, indeed, in a variety of gravitational and electromagnetic field situations is affected by the background field with a predictable alteration of the causal structure of space-time.

This phenomenon, it has been conjectured, gives rise to photons, depending on their direction and polarization, traveling with speeds exceeding the speed of light in modified Quantum Electrodynamics (QED) vacua. The explanation for this is that photon propagation in a background gravitational field is influenced by vacuum polarization; an effect by which the photon exists part of the time as a virtual e⁺ e⁻ pair. This virtual transition confers a size on the photon which is of $O(\lambda_c)$, where λ_c is the Compton wavelength of the electron. In a gravitational field the photon is therefore sensitive to an anisotropic space-time curvature and its characteristics of propagation may become curvature dependent, thus leading to the phenomenon of gravitational birefringence [5,6,7,8,9] or electromagnetic birefringence for an arbitrary anisotropic electromagnetic backgrounds [10,11,12,13]. Since electromagnetic and gravitational fields propagate in vacuum with the same constant Lorentz invariant speed c, when the vacuum is modified the speeds of propagation of particles and fields are modified as well. It is known that Finslerian space-times are anisotropic and that in them Lorentz invariance does not hold in general [14,15,16,17,18]. We will show in the work presented here that when the vacuum is modified by the presence of a non-vanishing torsionic potential, there is a possibility of birefringence.

PREPARATION OF PAINT BASED ON GUM ARABIC

Gum Arabic (250.0g) was dissolved in 800mls of distilled water (31%w/v) at room temperature for 2hrs with constant stirring until a homogeneous solution was obtained and this was subsequently filtered. Some 5mls of this filtrate was placed in twenty test tubes containing 0.02mls ammonia, 0.1mls formaldehyde, 0.2g kaolin and 0.2g CaCO₃. The pigment (1.156x10⁻⁴mole) was placed in each test tube. 1, ½, "÷ and ¼ mole equivalents each of ZnCl₂, MnCl₂, CuSO₄, Cr₂(SO₄)₃, FeSO₄ and NiCl₂ was placed in each test tube dropwise with stirring for 1hr at 35°C.

UV-Visible spectroscopy

2mls each of the prepared paints above were placed in test tubes and diluted with 8mls of ethanol. These were run in a uv-visible (M105 Camspec) spectrophotometer from 400-700nm at 5nm intervals using ethanol as blank.

The absorbances were read-up and the curves drawn using the Excel software, the respective wavelengths of maximum absorption, i.e., were then determined.

Tintometric colour measurements

The colours of the formulated paints were determined using the lovibond Schoffeld tintometer. 1ml of the sample was taken and dissolved in 50mls of ethanol. Some of this was transferred in to a cuvette, mounted in to the equipment and then matched with the primary colours i.e., Red(R), Yellow(Y) and Blue (B). The results are shown in Table 1

Viscosity measurements

The viscosities of the paints were measured with the Brookfield Synchro-electric viscometer (RVT) at a spindle speed of 100rpm. The results are shown in Figures 7-8.

Determination of the breaking stress of the coatings

The breaking stresses of the coatings were determined on a Motorized Automatic Monsanto tensometer. A shear load of 136.84kg was used. Twenty-five films each of the formulated coatings was formed on a white paper sheets with dimensions 15mm x 50mm and a thickness of 0.0035mm. The sheets were then immersed in the paint solutions, allowed to dry and then their new thickness measured. The difference between the thickness of the coated sheet and that of the uncoated one gave the thickness of the film alone. The breaking forces of the uncoated and the coated sheets were determined, the difference of which gave that of the film alone. The breaking stress was then calculated as the ratio of the breaking load (N) to the cross-sectional area (m²) of the film. Five measurements were made and the mean value computed. The results are as shown in Figures 9-10.

RESULTS AND DISCUSSIONS

The effect of salt concentration on the of the paints

The variations of with concentration of the salts are shown in Figures 1-6 and Table 1.

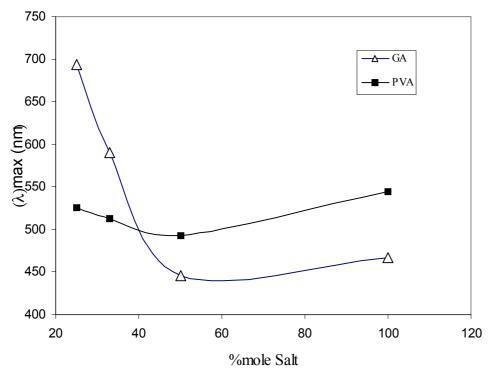


Figure 1: The variation of the wavelength of maximum absorption with ratio of the FeSO₄ salt

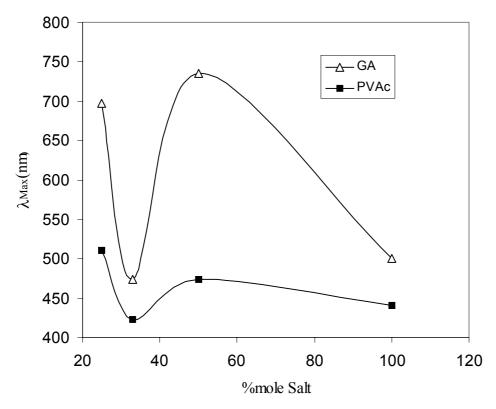


Figure 2: The variation of the wavelength of maximum Absorption with ratio of the CuSO₄ salt

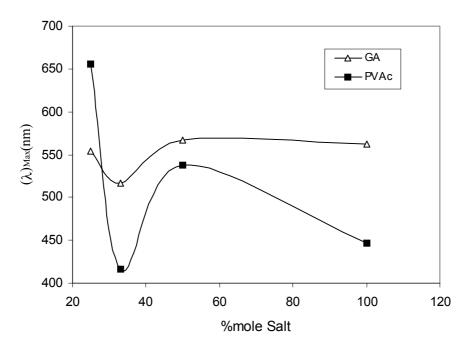


Figure 3: The variation of the wavelength of maximum absorption with ratio of the NiCl₂ salt

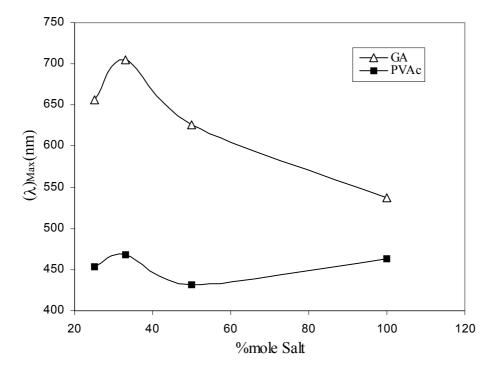


Fig. 4. The variation of the Wavelength of maximum absorption with ratio of the ZnCl2 salt

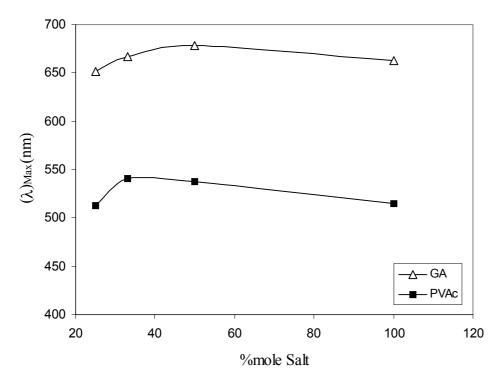


Fig. 5. The variation of the Wavelength of maximum absorption with ratio of the $MnCl_2$ salt

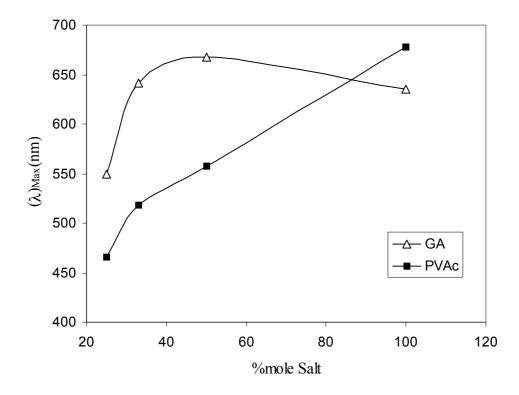


Figure 6: The variation of the wavelength of maximum absorption with ratio of the $Cr_2(SO_4)_3$ salt

Table 1: Uv-Visible and tristimulus colour components of the paints

Pigment %	Gum Arabic				PVAC			
	λ _{max} (nm)	В	Y	R	λ _{max} (nm)	В	Y	R
FeSO ₄ 100	467	5.50	46.0		545	10.0	4.0	5.0
FeSO ₄ 50	446	1.20	29.9		493		39.0	3.0
FeSO ₄ 33	590	2.60	69.0	3.0	513		60.0	3.0
FeSO ₄ 25	694	6.30	44.9		526		60.0	5.0
CuSO ₄ 100	500				441		6.4	37.0
CuSO ₄ 50	735	41.00	30.0		474		22.0	18.5
CuSO ₄ 33	474			78.0	423		42.0	19.8
CuSO ₄ 25	697				511		60.0	8.0
NiCl ₂ 100	562			35.6	447			55.9
NiCl ₂ 50	567			45.0	538		39.9	36.0
NiCl ₂ 33	517			51.0	416	10.0		73.5
NiCl ₂ 25	554			67.0	656			70.0
ZnCl ₂ 100	537		11.0	10.0	463		30.0	10.0
ZnCl ₂ 50	626		30.0	60.0	432		26.7	4.6
ZnCl ₂ 33	705		79.0	9.0	468		10.7	7.1
ZnCl ₂ 25	657	10.00		17.3	453		35.0	16.7
MnCl ₂ 100	663	7.30	13.0	10.0	515		44.0	20.0
MnCl ₂ 50	678	5.00	20.0	20.0	538		50.1	31.0
MnCl ₂ 33	667	1.00	51.0	8.0	541		10.0	5.2
MnCl ₂ 25	651	2.00	23.0	8.1	513		30.0	8.0
$Cr_2(SO_4)_3 100$	635				678		45.0	10.0
$Cr_2(SO_4)_3$ 50	668	2.90	30.2	8.2	558		41.0	10.0
Cr ₂ (SO ₄) ₃ 33	641		50.0	3.0	518	10.0		30.0
Cr ₂ (SO ₄) ₃ 25	550	4.00	75.0	30.0	466	30.0		20.0
Control	423	6.00	10.0		553		5.9	10.0

As could be observed, the shapes of the curves for the two binders in Figures 1 to 6, indicating the variations of

 λ_{max} with concentrations of the different salts are identical for the same salt, irrespective of the medium or binder. This, to a certain extent, is indicative that the nature of the complex formed between the pigment and the salts are the same irrespective of the binder and that the binder is not

taking part in the reaction. The marked differences in λ_{max} between the same salt/pigment in the two binders will have to do with the well-known phenomenon of solvochromism [Rao, 1975]. It could be explained from the differences in the polarities of the two binders. Obviously GA has higher polarity rating than PVAc even from their aqueous solubility profiles. In most of the cases the GA paints, recorded higher than the corresponding PVAc paints. It was reported elsewhere [Oforghor, 2003] that the paints' visible-spectra, are not sharp, as should be expected of metal complex dyes generally. The basic hues of the same family did not change contrastingly as could be expected from the often wide margins between their's.

In figures 1 to 3; representing the paints derived from Fe²⁺, Cu²⁺ and Ni²⁺ salts respectively, the λ_{max} of the 25% mole salt concentrations for both binders recorded the highest bathochromatic shifts. It is also seen that with Cu²⁺ and Ni²⁺ salts, the 33% mole equivalent concentrations gave paints with the least λ_{max} irrespective of the binder. The λ_{max} then rose at the 50% mole equivalent salt and

then fell again. The λ_{max} 's for Cu^{2+} salt in GA binder with increasing salt concentrations are: 500, 735,474 and 697 nm respectively, whereas the corresponding values for the PVAc binder are: 441,474,423 and 511 nm respectively. The values for the Ni²⁺ salt in GA are: 562, 567, 517 and 554nm and the corresponding values for the PVAc binder are: 447, 538, 416 and 656 nm respectively. Zn²⁺ and, to a certain extent, Mn2+ salts gave paints that recorded highest bathochromatic shifts at 33% mole equivalent concentration in all the binders. The's recorded for Zn²⁺ in increasing salt concentrations in GA binder are: 537, 626, 705 and 657nm while the corresponding values for the PVAc binder are: 463, 432, 468 and 453nm respectively. The corresponding values for Mn²⁺ in GA are: 663, 678, 667 and 651nm while in PVAc the values are: 515, 538, 541 and 513 nm respectively. It should be pointed out that the

 Mn^{2+} paints indicated the least change in λ_{max} . For the paints containing Cr^{3+} salts, the 25% mole equivalent salt concentration gave the lowest (hypsochromic) shift than

the others. Thereafter for the PVAc binder, the increased with the concentration of the salt, whereas for the GA binder, the λ_{max} maximum was obtained at the 50% mole concentration and then slightly decreased afterwards. These results, together with the Tristimulus colour components of the paints indicate that the colour obtained from these salts varied with the concentration of the salts.

Effect of salt concentration on the viscosity of the paints

The viscosity of the GA control, that is, the unsalted Gum Arabic paint is 2.00x 10⁻² Ns/m. As can be seen in Figure 7, the viscosity virtually increases above the control only at the higher concentration of the salts. The average viscosity of the paints in MnCl₂, ZnCl₂ and to a certain extent NiCl₂ did not change significantly with salting. In this group it was only the viscosity of 100% mole equivalent concentration of NiCl, that increased significantly to 3.00 x10⁻² Ns/m. However, for FeSO₄, CuSO₄ and Cr₂(SO₄)₃ the viscosity of the paints increased with the salt concentrations. The viscosity of the CuSO₄ containing GA paints increased to 3.00 x10⁻²Ns/m for the 25%, 33% and 100% mole equivalent concentrations it was only the viscosity of the 50% mole equivalent concentration that was significantly lower; 1.5 x 10⁻² Ns/m. FeSO₄ based paint increased from 2.00x 10⁻²Ns/m for the 25% mole equivalent concentration to 3.50 x 10⁻² Ns/m for the 100% mole equivalent concentration. The most significant viscosity increase is recorded for the Cr₂(SO₄)₃ salt whose value increased from 2.10 x 10⁻²Ns/m for the 25% mole equivalent to infinity for the 100% mole equivalent concentration. However, as pointed out earlier, Cr3+ was reported to precipitate GA [Mantel, 1947; 1954; Whistler et al, 1953; Stutz et al, 1990].

The PVAc control has a viscosity of 2.10x10⁻² Ns/m, generally, all the salted paints based on PVAc exhibited higher viscosity readings. The viscosity of these paints can be grouped into three. In the first group with Cu²⁺. Fe²⁺ and Ni²⁺ the viscosity shows a minimum for the 50% mole equivalent concentration. These minimum viscosities are 2.00×10^{-2} , 2.5×10^{-2} and 2.3×10^{-2} Ns/m respectively. As can be seen in Figure 8, the maximum viscosities of these salts are attained at the lowest concentrations being 25% mole equivalent concentration. They are 3.00x10⁻² Ns/m for the Fe^{2+} , $3.5x10^{-2}$ Ns/m for the Ni^{2+} and $3.7x10^{-2}$ Ns/m for the Cu²⁺ salts respectively. In the second group with the Cr³⁺ salt, the viscosity increases with the salt concentration consistently except for the viscosity at the 33% mole equivalent. The viscosities achieved by the Cr³⁺ salt at 100, 50, 33 and 25 %mole equivalent concentrations are 3.00x10⁻², 2.50x10⁻², 2.9x10⁻² and 2.00x10⁻² Ns/m

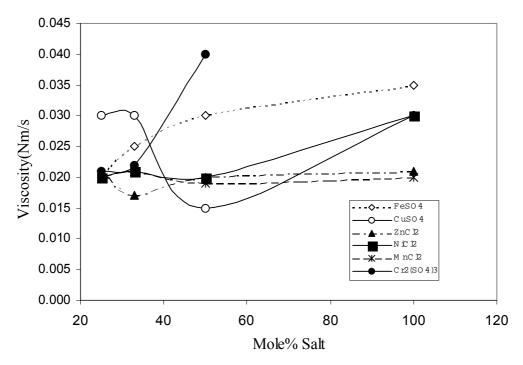


Figure 7: The viscosity for the Gum-Arabic paints in different salts

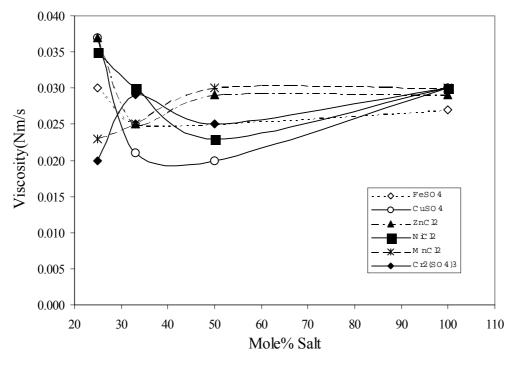


Figure 8: The viscosity of the PVAc Paints in different metallic salts

respectively. In the third group is Mn^{2+} salt, which shows increasing viscosity with concentration up to the 50% mole equivalent and then stabilize. The viscosities at 100, 50, 33 and 25 %mole equivalent concentrations are $3.00x10^{-2}$, $3.00x10^{-2}$, $2.5x10^{-2}$ and $2.3x10^{-2}$ Ns/m respectively.

Viscosity generally is dependent on the molecular weight and degree of esterification, concentration, pH and composition of the solution. Addition of salts of univalent cations, e.g. NaCl decreases the viscosity, caused by the suppression of charges on -COOH. But soluble salts of di- and trivalent cations show opposite effect. The viscosity increases in a regular fashion with the addition of Ca²⁺, Ba²⁺, St³⁺ and similar cations [Lotcker *et al*, 1946]. This observation has been ascribed to a bridging effect between suitably positioned -COOH groups, although it is likely that other effects as well are involved. Trivalent, metal ion salts i.e., Cr³⁺ caused precipitation of Gum Arabic [Mantel 1947, 1954; Whistler *et al*, 1953; Stutz *et al*, 1990].

In polymers that have highly branched stiff coiled configuration, there is this interchain hydrophobic bond behaviour. In dilute resin solutions with little interchain behaviour, added metal ions are effective in reducing the viscosity by bonding to the polymer (resin) and reducing the repulsive segment — solvent forces. At higher concentrations, however, where there is extensive hydrophobic bonding of side chains, added salts has no effect on solution viscosities; the hydrophobic bonding being stronger than the resin — electrolyte forces that occur at dilute concentrations. Viscosity increase can apparently result from a combination of H — bonding and electrostatic effect.

Effect of salt concentration on the breaking stress of the formulated coatings

Shown in Figs. 9 and 10 are the breaking stress of the PVAc paint and GA pain in different salts respectively. It is observed that the breaking stress of the Gum Arabic binder control (7.33 x 10⁵ N/m²) is higher than all the salted coatings from Gum Arabic except the coating with 25% mole equivalent MnCl₂. For the PVAc binder, the control containing only the unsalted pigment was about the least in breaking stress with (1.25 x 10⁵ N/m²) among its salted coatings. As seen in Figure 9, the breaking stress characteristics of the PVAc pigments can be grouped into three: One group, with salts of Cr³⁺, Zn²⁺ and Mn²⁺, recorded their maxima at the 33% mole equivalent salt concentration. The second group, with Ni2+ and Cu2+salts, recorded their maxima at the 50% mole equivalent concentration. It is interesting to note however, that the minimum breaking stress for Ni²⁺ salt occurred at 33%mole

equivalent, whereas the breaking stress of the Cu²⁺ salt decreased with decreasing salt concentration. The third group has only the salt of Fe²⁺; it recorded its least stress at 33% mole equivalent, which thereafter increased with increasing salt concentration without indicating a maximum. However, with the exception of the salts of Fe²⁺ and Cu²⁺ the breaking stresses of the other pigments decreased with increasing salt concentrations after their maxima. The order of maximum stress for the salts each at its best is: $Fe^{2+}(100\%)$: $[7.9x10^5N/m^2] > Ni^{2+}(50\%)$: $[6.2 \times 10^5 \text{N/m}^2] > \text{Cr}^{3+}(33\%)$: $[4.6 \times 10^5 \text{N/m}^2] > \text{Zn}^{2+}(33\%)$ and $Cu^{2+}(50\%)$: [4.3x10⁵N/m²] > Mn²⁺(33%): [3.7x10⁵N/m²]. The correlation between viscosity and breaking stress readings is strong in some cases as compared to others. For Cr³⁺ in PVAc binder for example, with decreasing salt concentrations the viscosity readings are: 3.00x10⁻², $2.50x10^{-2}$, $2.90x10^{-2}$ and $2.00x10^{-2}$ Ns/m and the corresponding breaking stresses are: 2.1x10⁵, 1.74x10⁵, 4.64×10^5 and 4.06×10^5 N/m². It could be seen that, just as the viscosity readings increased in two stages so also was the breaking stresses, in all cases a strong correlation with positive slope could be observed. The Cu²⁺ salt in PVAc solution, however, gave results, which showed correlation with negative slope if the 33% mole equivalent concentration is disregarded. The breaking stresses with decreasing concentration of the salt are: 3.92x10⁵, 4.29x10⁵, 3.33x10⁵ and 3.11x10⁵ N/m², the corresponding viscosity readings are: $3.00x10^{-2}$, $2.00x10^{-2}$, $2.10x10^{-2}$ and $3.70x10^{-2}$ Ns/m respectively. Similar observation could be made with respect to the Ni²⁺ salt in PVAc, if the 33% mole equivalent concentration is disregarded. The breaking stresses with decreasing salt concentration are: 3.9x10⁵, 6.20x10⁵, 1.77x10⁵ and 2.75x10⁵ N/m². The corresponding viscosities are: 3.00x10⁻², 2.33x10⁻², 3.00x10⁻² and 3.5x10⁻² Ns/m respectively. Similarly, the Zn²⁺ salt in PVAc shows the same trend, if the 100% mole equivalent concentration is disregarded. The breaking stresses with decreasing salt concentrations are: 1.07x10⁵, 3.20x10⁵, 4.3x10⁵ and 2.10x10⁵ N/m². The corresponding viscosities are: 2.90x10⁻², 2.90x10⁻ 2 , 2.50x10⁻² and 3.70x10⁻² Ns/m respectively. Whereas Cr³⁺ and Mn²⁺ salts do not indicate a very strong correlation, however, they all indicate the negative slope types.

The trends of the Gum Arabic paints with respect to the breaking stress can be grouped into two. The first group; with Mn²⁺, Cu²⁺, Zn²⁺ and Cr³⁺ salts recorded a maximum at 25% salt equivalent concentration. With the exception of the Zn²⁺ which stabilises somehow, the breaking stresses of all the other salts in this group decrease with increasing salt concentration. The second group has the salts of Ni²⁺ and Fe²⁺; they recorded their maximum breaking stresses at the 50% mole equivalent salt concentration. The order of maximum breaking stresses

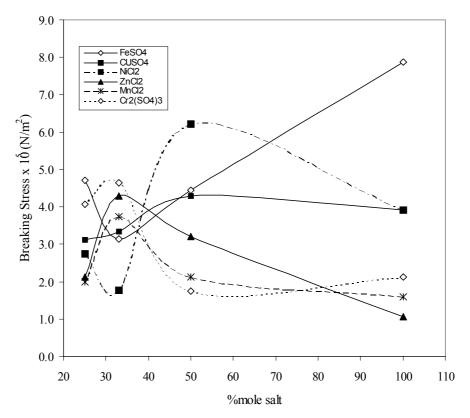


Figure 9: The breaking stress of the PVAc paint in different salts

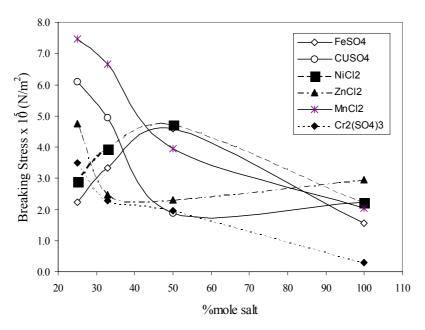


Figure 10: The Breaking Stress of the Gum Arabic Paints in different salts

for the salts each at its best is: $Mn^{2+}(25\%)$: $[7.5x10^5N/m^2] > Cu^{2+}(25\%)$: $[6.1x10^5N/m^2] > Zn^{2+}(25\%)$: $[4.8x10^5N/m^2] > Ni^{2+}(50\%)$: $[4.7x10^5N/m^2] \sim Fe^{2+}(50\%)$: $[4.6x10^5N/m^2]$.

There is strong correlation between the viscosity readings and breaking stresses with a salt such as Cr^{3+} . This salt showed that the higher the viscosity the lower the breaking stress and vice versa. The viscosity readings with decreasing salt concentrations are: infinity, 4.00×10^{-2} , 2.2×10^{-2} and 2.1×10^{-2} Ns/m whereas the corresponding breaking stress readings are 0.308×10^{5} , 1.95×10^{5} , 2.28×10^{5} and 3.48×10^{5} N/m² respectively. The Fe²+ salt also gave similar trend if not for the breaking stress of the 100% mole equivalent concentration. The breaking stresses with decreasing salt concentration of Fe²+ are: 1.56×10^{5} , 4.58×10^{5} , 3.33×10^{5} and 2.50×10^{5} N/m² the corresponding viscosity readings are: 3.5×10^{-2} , 3.00×10^{-2} , 2.50×10^{-2} and 2.00×10^{-2} Ns/m respectively. Similar observation could be made for the Ni²+ salt.

The mechanical properties of a surface coating depend strongly on many factors, two of the most important are: (i) the lengths of the segments between cross-links, and (ii) the glass transition temperature (T_{-}) of the cross-linked resin. The term cross-link density (ČLD), is sometimes used to express the length between a cross linked segments. It is defined as the number of moles of elastically effective network chains per cubic centimetre of film. The T_{σ} of the polymer segments between cross-links is governed by the chemical structures of the resins and the cross-linking agents and by the ratio of these components [Stutz et al, 1990]. T_g increases as cross-link density increases and decreases with increasing proportion of dangling ends, i.e., chain segments with unreacted crosslink sites. This adversely affects the mechanical properties of the coating [Zewo et al, 1992]. It is most likely that the observed increase in the breaking stress with decreasing viscosity will be related with the cross-link density. This is because the intermolecular interactions between the chains of the binder could be affected by the chelation.

CONCLUSION

The formulated paints containing the pigment showed evidence of complexing with the metals in the two binders used. This evidence was witnessed by the changes in the colour of the paints immediately the salts were added. It was also seen that, though the main hue of the paint was maintained but the actual colours generated by altering the ratios of the salts were different as was indicated by the changes in the wavelengths of maximum absorptions and the tristimulus colour compositions of the paints. The

viscosities of the freshly prepared paints were defined by the ratios of the salts added. In most cases viscosity increased with the decrease in the concentration of the salt. The breaking stress of the coatings, which is directly proportional to the film strength also varied with the concentration of the salts. The PVAc paints containing Cr³⁺, Zn²⁺, and Mn²⁺ salts gave better results in 33% mole equivalent concentration or 1:3 ratio of salt to pigment. Ni²⁺ and Cu²⁺ gave their best results in 50% mole equivalent concentration or 1:2 ratio salt-to-pigment and were worst in 33% mole equivalent concentration. The next group has Fe²⁺ salt, which produced its worst result in 33% mole equivalent concentration, thereafter the breaking stress increased with increasing salt concentration. The Gum Arabic paints were mostly best in 25% mole equivalent concentrations or 1:4 salt-topigment ratio. Among the salts only Ni²⁺ and Fe²⁺ produced their best results in 50% mole equivalent concentration. It was also obvious that generally breaking stress was inversely proportional to the viscosity.

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