

## COPPER WIRES COATED WITH ENAMEL

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### ABSTRACT

The polyvinyl formal (PVF), which was prepared by reacting polyvinyl acetate (PVA) with formaldehyde and acetic acid (HAC) for one day at 70 °C, was reacted with phenolic resin (PR) for 1/2 hr at room temperature in order to produce specified enamels. The electrical wires were then coated with 0.14 mm thick enamels, which contain 80% PVF in the PVF/RP mixtures for 15 minutes at 150 °C, in order to produce required properties, such as 2.3 Kg/cm peeling stress, 20 Kg/cm<sup>2</sup> shear stress, 32 million ohm impedance and 5500 volts breakdown voltage. Additions of 15% epoxy resin, as additive, to enamels were also studied, by evaluating chemical, mechanical, and electrical properties of the produced coated enamels.

### INTRODUCTION

Electrical insulation of copper or brass or aluminum wires, which contain mainly hydrocarbon adhesives with very low content of oxidizing agents such as oxygen, chlorine, sulphur to reduce or minimize flow of electrical current, were studied by different investigators.<sup>[1-3]</sup> These hydrocarbon adhesives must withstand chemical, mechanical, and electrical stresses such as pressure, temperature, tensile stress, flexural stress, electrical resistance, abrasion resistance, X-ray, UV light, hardness, adhesion strength, cohesion strength, high voltage, high current, moisture.<sup>[1-5]</sup>

There are many forces between metallic surfaces and adhesives such as cohesion and adhesion forces. The bonding between the metal surfaces and the enamel coating adhesives are responsible to hold them together such as hydrogen, electrostatic, van der Waals, polar, ionic bonding.<sup>[3-6]</sup> The enamel film is stuck to copper wire due to polar groups. Coating can be carried out in vapour, liquid, or solid phase depending on the types of adhesive and adherents. Impurities on the surfaces, such as dust, water, air, and influence adhesion strength. Correct controls of solvents, oxygen, heat, catalysts, water, ...etc. influence adhesion properties due to the thermodynamic properties of the adhesives and impurities.<sup>[6,7]</sup> Cleaning the surfaces of the metals by different chemicals, in order to remove the contaminants of dust or metallic impurities from the surfaces, is required before using enamels.<sup>[8-10]</sup>

Reactions between different chemicals used to obtain required enamels, such as polycondensation reaction, poly addition were carried out at different conditions. There are many types of resins used to coat metallic surfaces, such as epoxy resin, phenolic resin, polyvinyl acetate, polyvinyl alcohol. Polyvinyl acetate, for example, reacts with isocyanates, dialdehydes, melamines in order to produce adhesive for electrical insulations.<sup>[9-12]</sup> During surface preparation, acid treatments or alkaline treatments were used depending on the types of metals used.<sup>[1,2]</sup>

Curing of the mixture is conducted in order to obtain required enamels, by using different operating conditions, such as temperature, pressure, time, catalyst, solvent. Different solvents should be tried, such as acetic acid, ethanol, benzene, toluene, hexane, in order to obtain homogeneous solution.<sup>[11,13]</sup> It is required for the prepared enamel to withstand high voltage, before breakdown occurs, in order to be used for electrical insulation. Polymerization mechanism influence the type of enamels to be suitable for electrical appliance.<sup>[14-15]</sup> Rates of polymerization influence electrical resistance of enamels.

The purpose of this work is to find suitable enamels to be used to coat copper wires and to withstand chemical, mechanical, and electrical stress by evaluating the operating conditions before breakage of the enamels occur.



## EXPERIMENTAL

The experimental procedure involves the preparation of polyvinyl formal (PVF), preparation of enamel, adding an epoxy resin to the enamel, coating the copper wire with enamel, and complete analysis of the suitability of the produced enamel for electrical insulation by measuring the chemical, mechanical, and electrical properties.

Preparation of polyvinyl formal (PVF) was conducted from the reaction of 76 gm of polyvinyl alcohol or polyvinyl acetate with 828 gm of acetic acid and with 67 gm of 40% formaline using sulphuric acid as catalyst and water as solvent. This reaction was carried out for one day at 70°C, with continuous agitation. The stoichiometric equation of chemical reaction indicates that 13.8 gmole of acetic acid, 0.9 gmole of polyvinyl acetate, and 0.9 gmole formaline were reacted. During the reaction, the weight of PVF produced at different time intervals were evaluated and found out to increase till PVF reaches a value of 650 gm after 24 hrs. This reaction gives constant quantity of produced water, i.e.: 96 gm after 24 hrs.

Phenolic resin (PR) was then reacted with produced PVF at room temperature using acetic acid as a solvent to give specified enamel. Other solvent, such as benzene, toluene, ethanol, naphtha, were tried but failed to give homogenous solution. Various amounts of epoxy resins, (i.e.: 5-20%), were added as additives to the prepared enamel in order to investigate the improvements of enamel properties. The reaction was completed within 5 mins as indicated by homogenous solution. Higher concentrations of the epoxy resins gave undesirable results.

The final stage, after treatments of copper wires with acetic acid for 1/2 hr at room temperature, the copper wires were immersed for a few seconds in different concentrations of PVF-PR enamels. This operation was repeated many times, about 15 times, in order to get different enamel thickness. The enamel was then put in oven at 150°C for 10 seconds in order to get rid of the moisture. It was found out that HAC treatments to copper took 30 mins. Immersions made pitches, which increase copper sheet roughness from 0.5 to 1.0  $\mu\text{m}$ , which gave higher adhesion of enamels on copper sheets. The enamel

thickness increases with the number of immersions of copper wires in enamels up to four immersions. After that, the number of immersions have no influence on enamel thickness.

The physical properties of enamels were evaluated according to ASTM and IEO standards by measuring density, electrical conductivity, viscosity, and PH of the enamels. Similarly mechanical properties of enamels were evaluated such as elongation, shear stress, peeling, penetration. Copper sheets of 15 cm \* 2.5 cm with area of adhesion 6.25 cm<sup>2</sup> was used for measuring shear stress and elongation. But for measuring peeling, the area of adhesion of 56.25 cm<sup>2</sup> was used. Electrical properties of enamels were also measured by impedance, and breakdown voltage up to 5500 volts.

## RESULTS AND DISCUSSION

The weight ratio of polyvinyl acetal to polyvinyl acetate to polyvinyl alcohol was 82/12/6. Molecular weight of polyvinylformal is 650 g. This gives 13.8 gmole acetic acid, 0.9 mole polyvinyl acetate and 0.9 gmole formaline, which were reacted using 4 ml of 98% sulphuric acid as catalysts at 70°C for one day, in order to produce 1 gmole of polyvinyl formal, 5.32 gmole of water and 5.8 gmole of oxygen. The reaction was completed as indicated by the absence of turbidity due to the absence of water. Crystallization of the polyvinyl formal from the solution was obtained. Acetic acid was found to be best solvent, which can dissolve phenolic resin. Other solvents, such as ethanol, toluene, benzene, gave unsatisfactory results.

The prepared polyvinyl formal (PVF) was then reacted with phenolic resin (PR) using acetic acid solvent in order to get homogenous solution after 30 minutes at room temperature. Many additives were used to improve the quality of adhesive such as polyvinyl chloride, melamine resin, isocyanate resin, polyester resin, epoxy resin and it was found out that only epoxy resin with weight ratio of 0.05 to 0.25 gave homogenous solution of PVE/PR mixture after 5 minutes at room temperature. Different experimental tests of the percentages of the polyvinyl formal in the enamel mixture were investigated by measuring the adhesive power of the enamel.



There is a big decrease in pH, density, viscosity index and electrical conductivity with and without epoxy resin additive; with increasing percentages of polyvinyl formal in the enamel as shown in fig. (1). The pH and density were higher for enamels without additives than those with additives. While the viscosity index and electrical conductivity were lower for enamels without additives than those with additives, due to the structural configuration and properties of the components in the enamels.

The number of immersions of wire in enamels depend on the viscosity and density of the enamel in order to obtain required electrical conductivity of the enamels which is important factor in insulation due to current fluctuations passing through the wire.

The effects of temperature up to 300°C, and curing time up to 30 minutes on the peeling stress at different weight ratio of polyvinyl formal in the enamel; i.e: 0.5, 0.6, 0.7, 0.8; with and without additives are reported in fig. (2). This figure shows the maximum peeling stress at 150°C with 15 minutes curing time. As peeling increases with time and temperature, till it reaches maximum value and then peeling decreases with temperature and curing time. The best suitable quantity of additives used to get good enamel properties was 15% by weight epoxy resin. Polyvinyl formal is more plasticizing than phenolic resin, therefore higher weight ratio of polyvinyl formal gives higher peeling stress.

There is slight effect of temperature on shear stress up to 100°C. After that temperature, a big increase of shear stress is observed up to 200°C. Above 200°C, there is slight effect of temperature on shear stress, as shown in fig. (3). The effect of curing time on shear stress was found to be almost linear. Therefore, the optimum condition of temperature is 150°C and curing time is 15 minutes. Shear stress increases with percentages of polyvinyl formal, because polyvinyl formal controls adhesion forces and phenolic resin controls cohesion forces and also because phenolic resin is rigid. Therefore 70% - 80% of polyvinyl formal in enamels was found to give better shear stress and peeling. The effect of epoxy resin on enamel was found to increase the shear stress in enamel with additives than without additives due to

plasticizing nature of epoxy resin. The additions of 15% by wt. epoxy resin gave good enamel properties, because the increase of shear stress (about 15% increase) was observed due to the plasticizing nature of epoxy resin. The results indicate good adhesion as shown by the breakages of metallic copper sheets instead of enamel adhesive materials between the sheets, because peeling stress measures adhesion forces between enamel and copper, while shear stress measures cohesion forces between enamel molecules.

The elongation of the enamel with and without additives decreases linearly with temperature and curing time as shown by fig. (4), due to cross-linking formation between the molecules at a high temperature and long curing time. The higher weight ratio of polyvinyl formal gives increase in elongation due to plasticizing properties of polyvinyl formal. The elongation results of enamels are within allowable ASTM and IEC specifications, i.e: 33%. The results indicate linear decrease of penetration (mm) with phenolic resin in enamels, since hardness indicates higher phenolic resin due to its rigidity and less flexibility while higher polyvinyl formal gives higher flexibility due to its plasticizing nature of polyvinyl formal.

The effects of enamel thickness on impedance for different weight ratio of polyvinyl formal in enamels are shown in fig. (5). The results indicate an increase of impedance (i.e: a higher decrease in electrical conductivity) with enamel thickness till it reaches maximum value at around 0.14 mm. thickness for 80% polyvinyl formal and after that, the effects of thickness on the impedance is slight. The results indicate that the higher percentages of polyvinyl formal, the higher impedance due to higher ionic radicals in phenolic resin as shown by the value of 33 Mega ohm impedance for 80% polyvinyl formal with 0.14 mm. thick.

The enamel coating was subjected to different voltages for different types of enamels and for different enamel's thickness, as shown in fig. (6). The maximum voltage, which allows (2 mA) to pass through that enamel, was measured. The results indicate that breakdown voltage increases with enamel thickness up to certain value. After that thickness has no effect on breakdown voltage.



At this level breakdown voltage remains constant with thickness. The higher the polyvinyl formal in the enamel will give greater value of breakdown voltage. Similarly the higher polyvinyl formal ratio shows the greater thickness of the enamel. Further thickness does not influence breakdown voltage due to high electrical resistance of polyvinyl formal in comparison with phenolic resin. The experimental results indicated for 0.14 mm. thickness of enamel containing 80% polyvinyl formal requires 5500 volts as breakdown voltage compared with recommended 2500 volts as breakdown voltage as given by IEC specifications.<sup>(2)</sup>

The effects of polyvinyl formal ratio at constant temperature, i.e. 150°C, and at constant curing time, i.e. 15 minutes on the shear stress, peeling stress, elongation, and penetration with and without epoxy resin additive were plotted in fig. (7). This figure indicates an increase in the shear stress, peeling stress, elongation and penetration with polyvinyl formal ratio. Penetration rates were lower without additives than with additives.

## CONCLUSIONS

The polyvinyl formal was prepared from the reaction of 76 gin of polyvinyl acetate, 800 gm of acetic acid, and 67 gm of 40% formaline using sulphuric acid as catalyst and water as solvent. Then polyvinyl formal was reacted with phenolic resin using acetic acid as solvent to produce enamel. Epoxy resin was added as additives to improve adhesive properties. Different percentages of polyvinyl formal were used in the enamels, such as 50%, 60%, 70% and 80%.

It was found out that 80% polyvinyl formal in the enamel with 0.14 mm. thickness gave best chemical, mechanical and electrical properties such as 20 Kg/cm<sup>2</sup> shear stress, 2.3 Kg/cm peeling stress, 25% elongation, 32 million ohm impedance, 5500 volts breakdown voltage. Adding 15% epoxy resin to the enamel improves certain properties. The results indicated that best enamel could be obtained with 80% polyvinyl formal at oven temperature of 150°C for 15 minutes curing time. After treatments of copper wire with acetic acid for ½ hr., the copper wire was immersed for few seconds in different

concentrations of enamels in order to be covered with required thickness.

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## SYMBOLS

PVF	= Polyvinyl Formal
PR	= Phenolic Resin
HAC	= Acetic Acid
ER	= Epoxy Resin
PVA	= Polyvinyl Alcohol
PVAC	= Polyvinyl Acetate
PVAL	= Polyvinyl Acetal



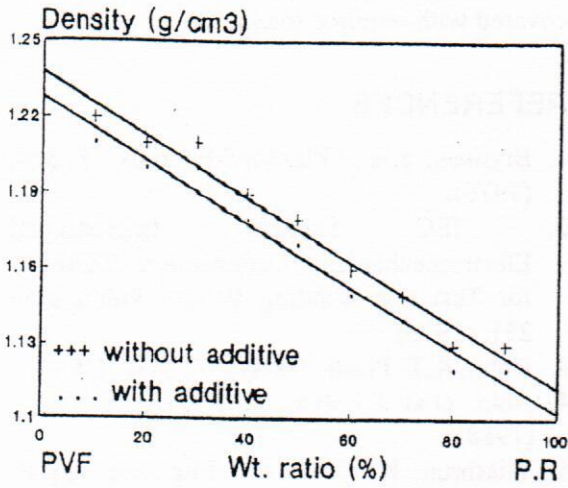


Fig. (1-a) Effect wt. ratio (PVF/P.R) on density

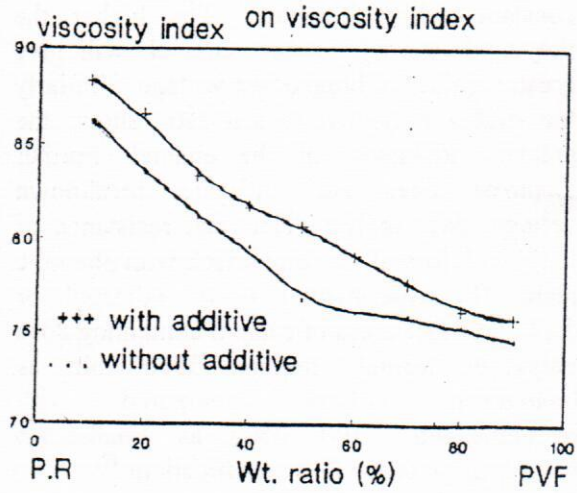


Fig. (1-d) Effect of wt. ratio (PVF/P.R) on viscosity index

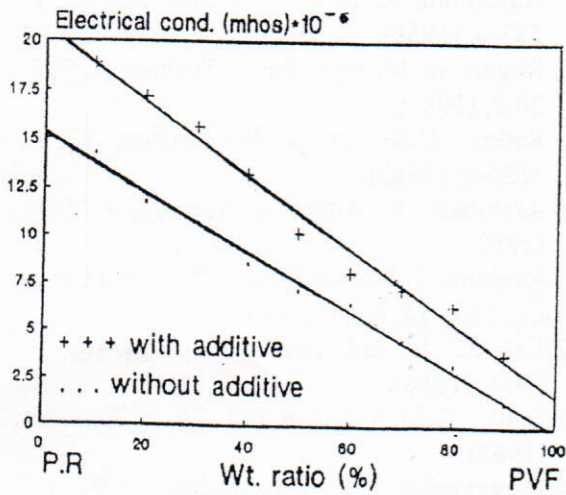


Fig. (1-b) Effect of wt. ratio (PVF/P.R) on electrical conductivity

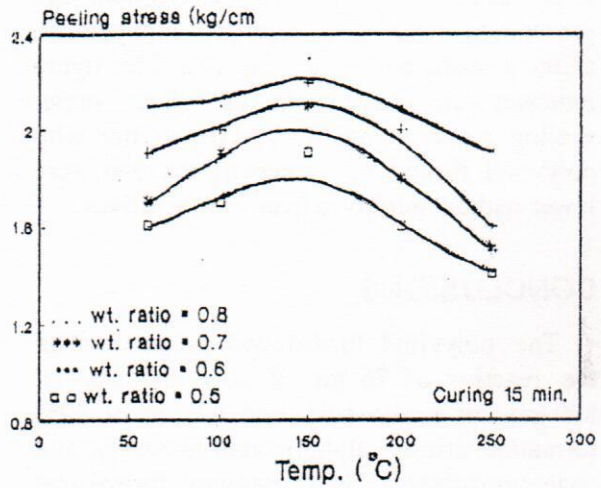


Fig. (2-a) Effect of oven temp. on peeling stress with additive

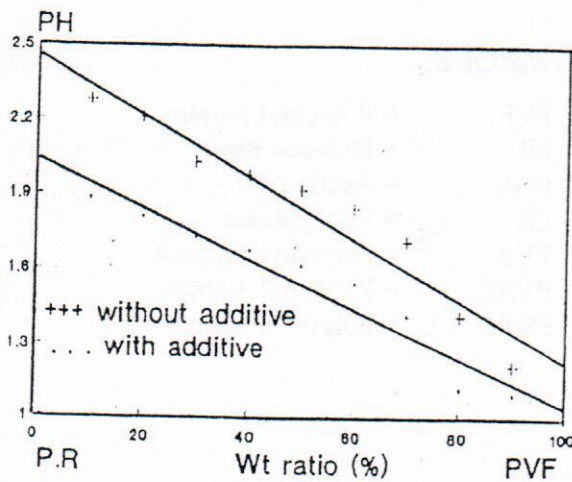


Fig. (1-c) Effect of wt. ratio (PVF/P.R) on PH

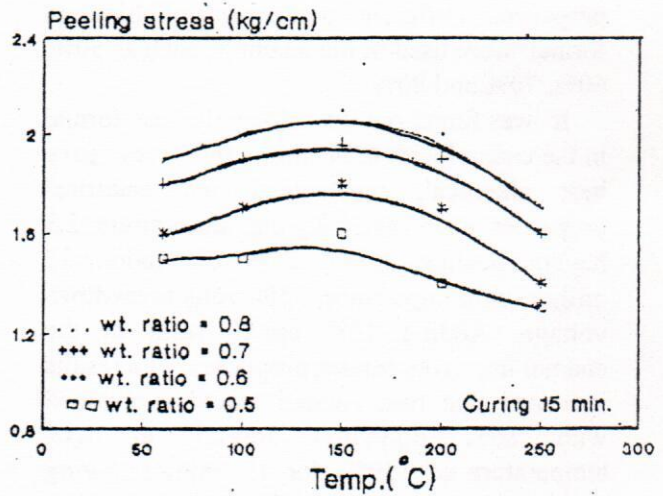


Fig. (2-b) Effect of oven temp. on peeling stress without additive

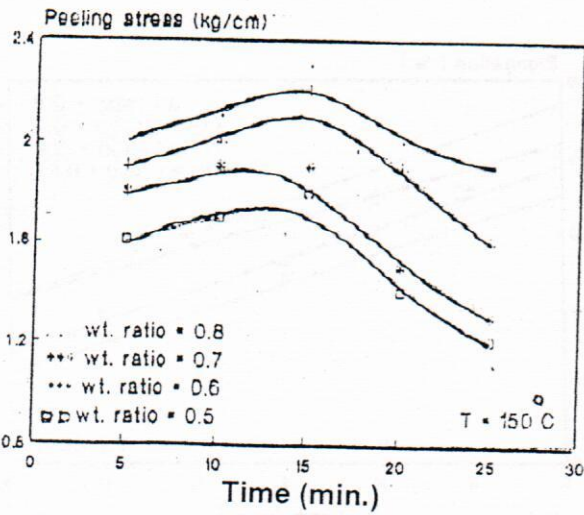


Fig. (2-c) Effect of time of curing on peeling stress with additive

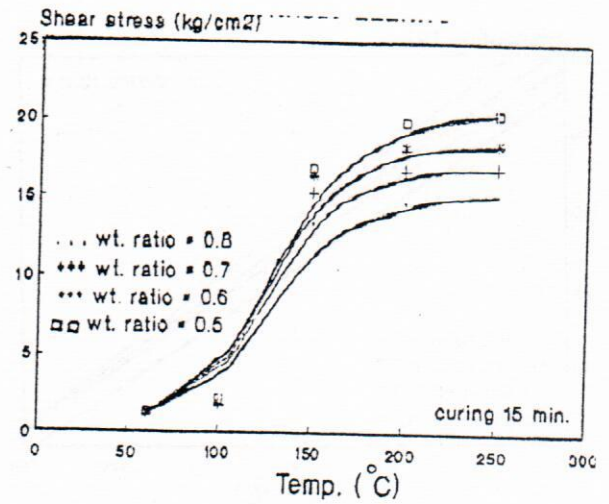


Fig. (3-b) Effect of oven temp. on shear stress without additive

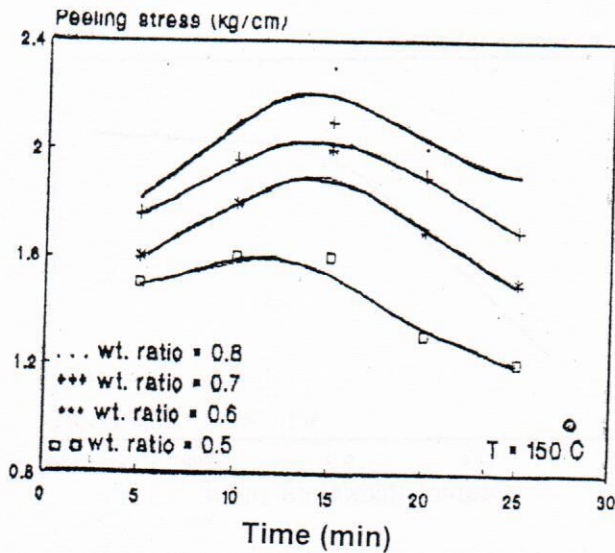


Fig. (2-d) Effect of time of peeling stress without additive

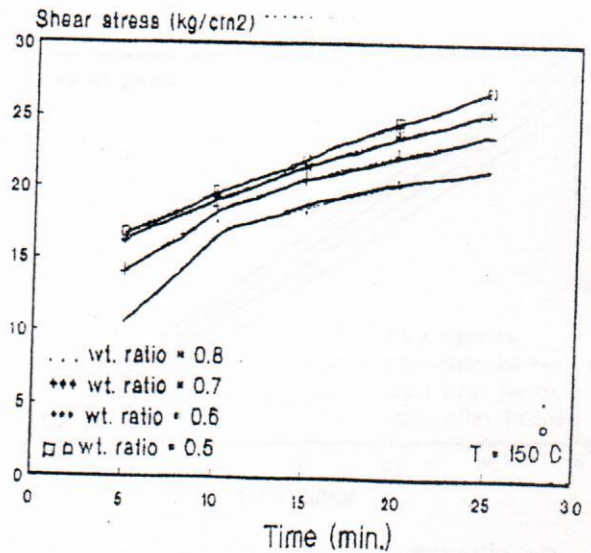


Fig. (3-c) Effect of curing on shearing stress with additive

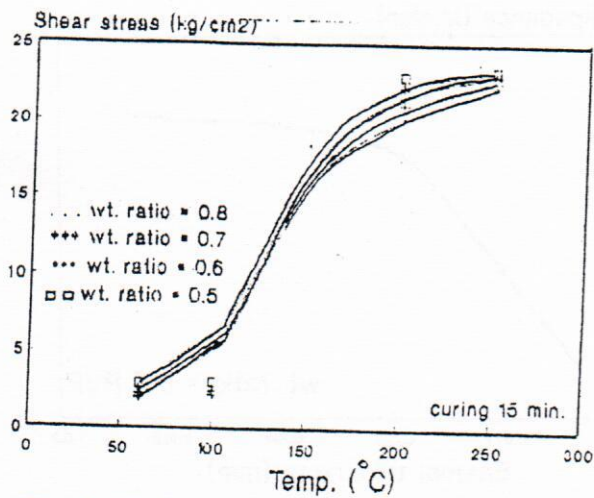


Fig. (3-a) Effect of oven temp. on shear stress with additive

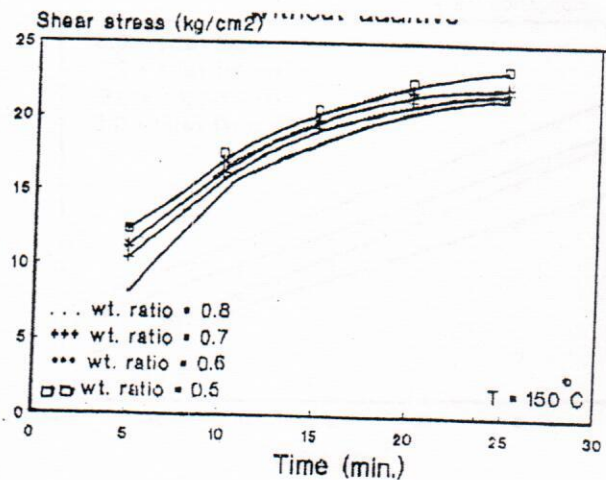


Fig. (3-d) Effect of time of curing on shear stress without additive



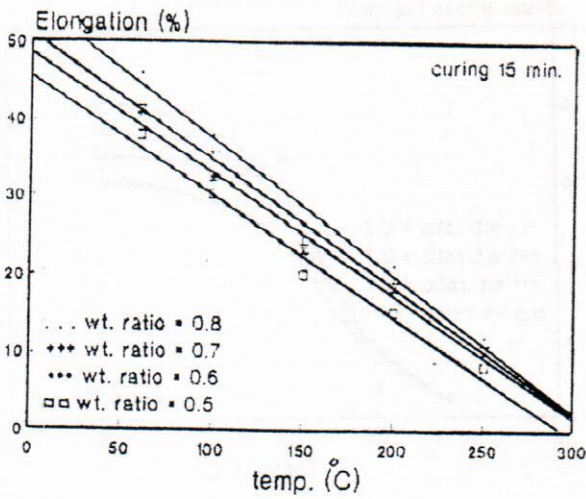


Fig. (4-a) Effect of oven temp. on elongation with additive

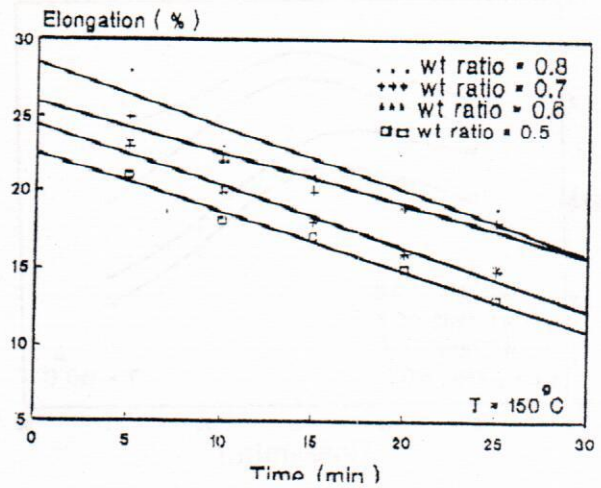


Fig. (4-d) Effect of time of curing on elongation without additive

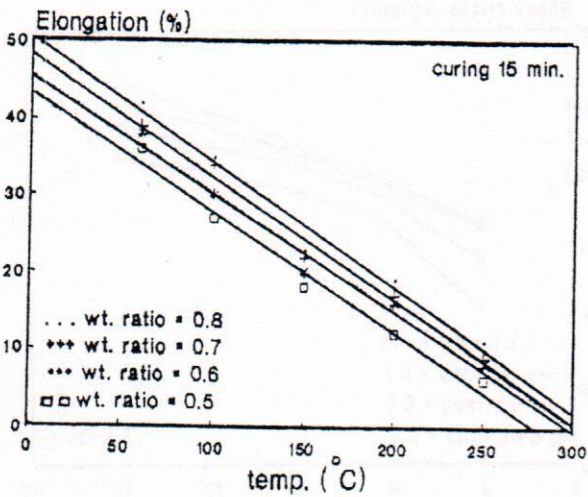


Fig. (4-b) Effect of oven temp. on elongation without additive

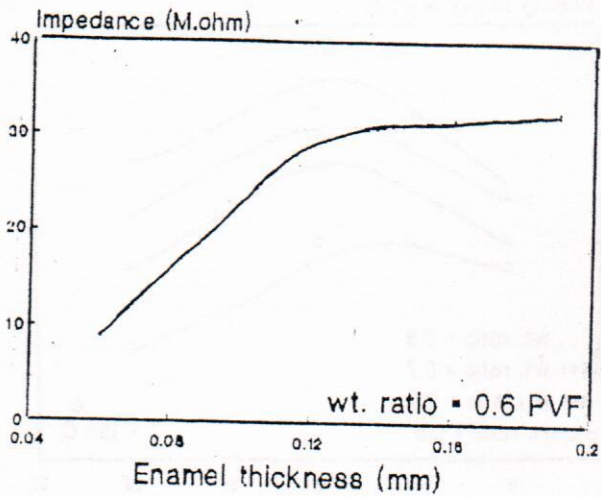


Fig. (5-a) Effect of enamel thickness on enamel impedance

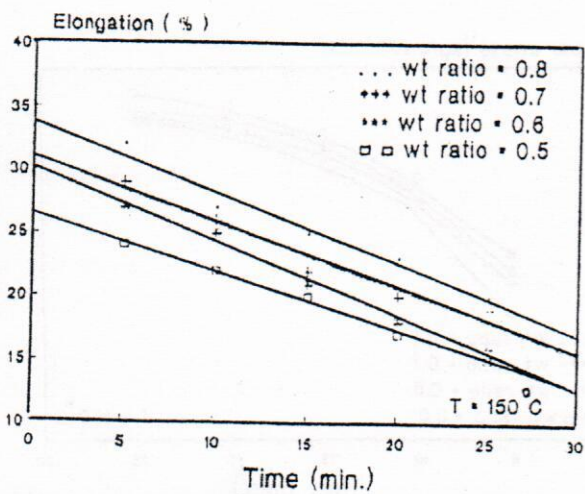


Fig. (4-c) Effect of time on elongation with additive

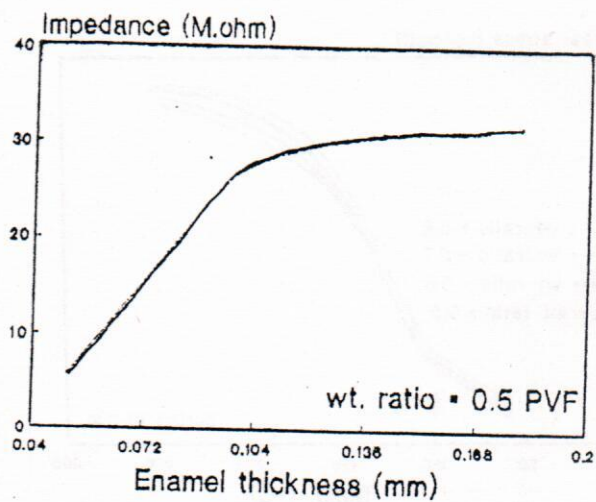


Fig. (5-b) Effect of enamel thickness on enamel impedance

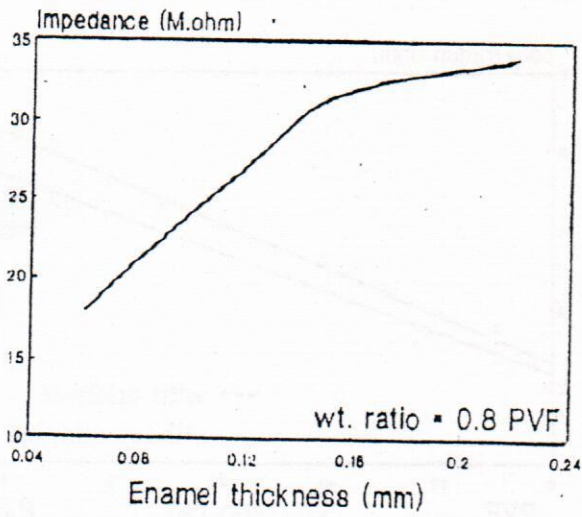


Fig. (5-c) Effect of enamel thickness on enamel impedance

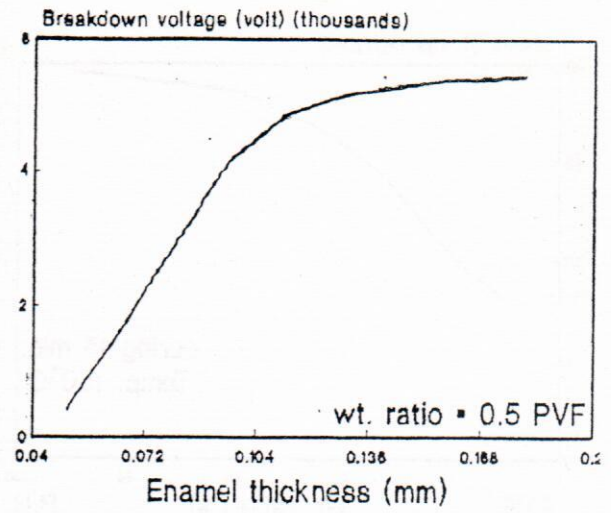


Fig. (6-b) Effect of enamel thickness on breakdown voltage

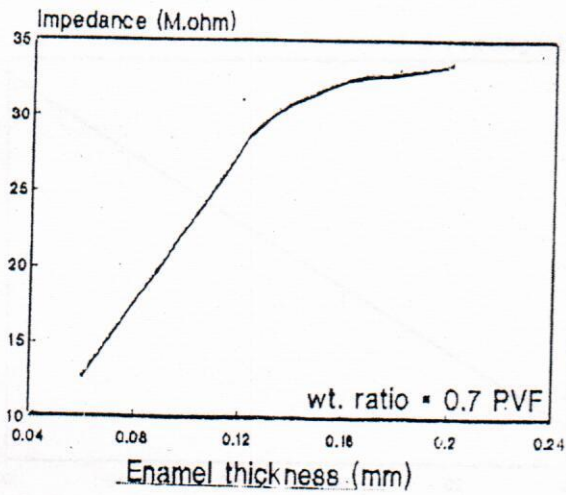


Fig. (5-d) Effect of enamel thickness on enamel impedance

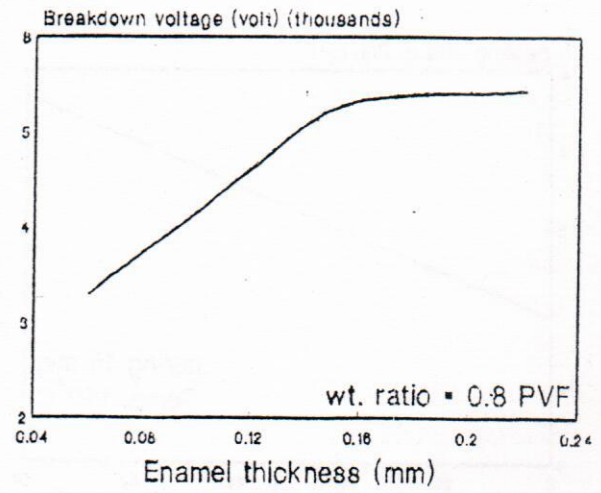


Fig. (6-c) Effect of enamel thickness on breakdown voltage

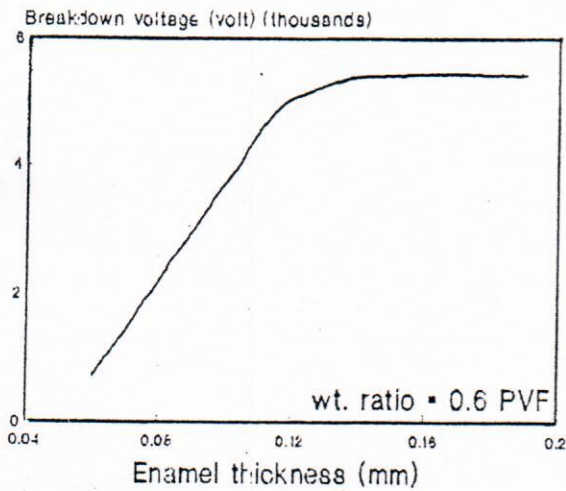


Fig. (6-a) Effect of enamel thickness on breakdown voltage

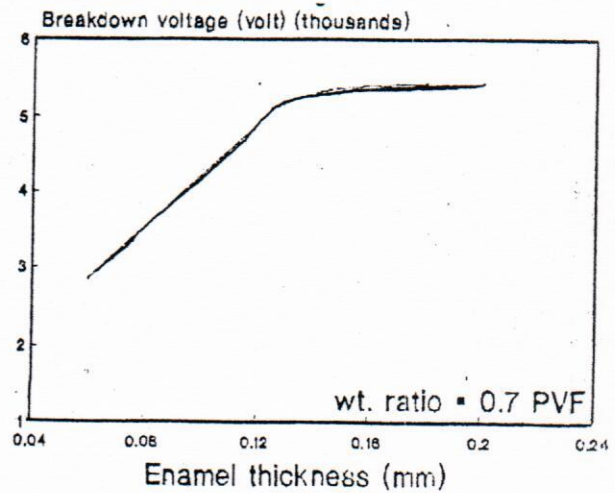


Fig. (6-d) Effect of enamel thickness on breakdown voltage



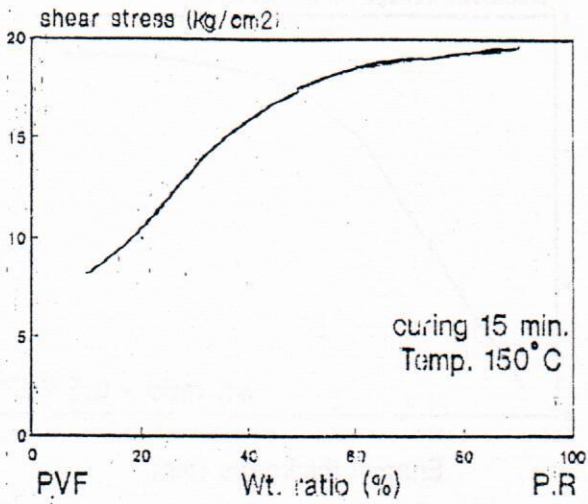


Fig. (7-a) Effect of wt ratio (PVF/P.R) on shear stress

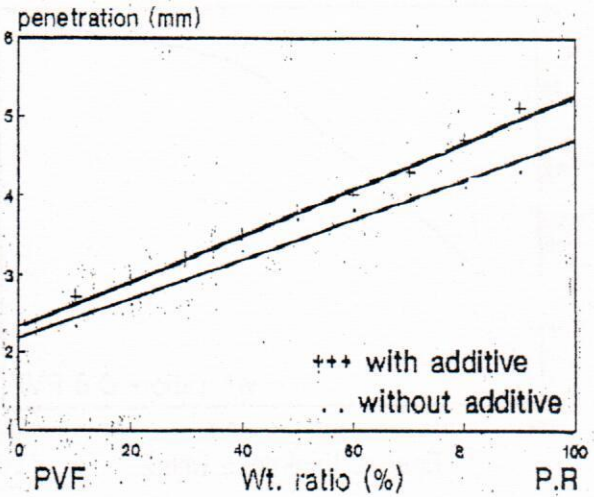


Fig. (7-c) Effect of wt. ratio (PVF/P.R) on enetration

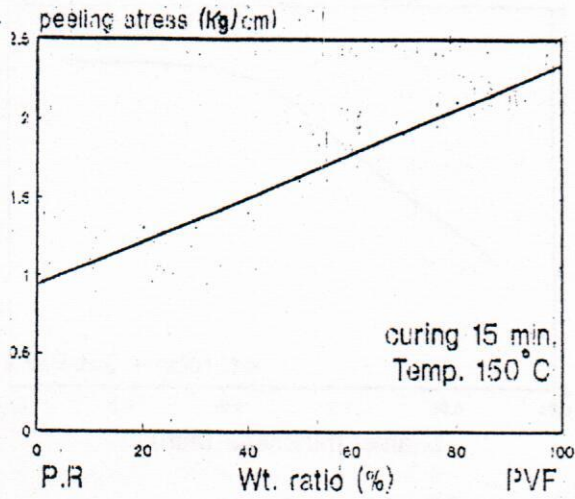


Fig. (7-b) Effect of wt. ratio (PVF/P.R) on peeling stress

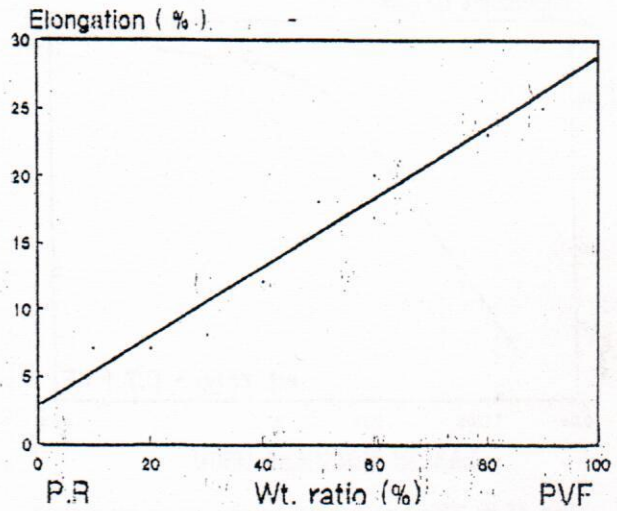


Fig. (7-d) Effect of wt. ratio (PVF/P.R) on elongation