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Preparation and Characterization of a New Composite Material from Wastes of Ceramic

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Abstract— In this work, we characterize the effects of temperature on a composite material comprising a matrix based on thermosetting unsaturated orthophthalic polyester resin (UPR) and reinforcing base powder waste Original porcelain ceramics industries. The results show that, generally, the new composite material is resistant to the temperature increase. This has been attributed to the effect of the load cling well to the resin. In addition, we noticed that for a large mass ratio of porcelain, mass loss due to the effect of the temperature decreases significantly. Indeed, incorporation of the powder in the porcelain orthophthalic polyester resin which has an amorphous structure gives the semi-crystalline material, in our study, the effect of the load increases the intensity of the peaks obtained.

Index Terms— UPR resin; Porcelain powder; humidity level; weight loss; Temperature.

I. INTRODUCTION

Composites combine excellent mechanical properties with low density. However, in terms of service, these materials are exposed to various stresses, thermal, chemical and mechanical, which can involve significant loss of property and compromise the integrity of structures [2].

Because of their low cost, the most commonly used binders are those based of unsaturated polyester resin (UPR) in most applications, the binder is a polyester prepolymer formulation general purpose unsaturated polyester_resin (UPR). These formulations are in the form of solutions consisting of 60 to 80 percent of the prepolymer dissolved in the copolymerizable monomers such as styrene and styrene-methyl methacrylate. During curing, the polyester prepolymer and monomer react through their unsaturated groups (double bonds). In the literature [3-4] three main monomers are employed for the synthesis of unsaturated polyesters: propane-1,2-diol (propylene glycol), the benzenedicarboxylic anhydride (phthalic anhydride) and the anhydride-purpose 2-enedioic acid (maleic anhydride).

The aim of this study was to investigate to thermal stability of new composite based on unsaturated polyester resin (UPR) and Porcelain Powder origillay from waste ceramics of city of settat in Morrocco. Knowing that this study was done to determine the possibility of using this charge from waste ceramic instead of silica that exhibits these last years an extra cost on society Hygimar . This study was carried out between the Faculty of Science and Technology of settat and Hygimar Company moved to the city of benslimane (Morocco). We noite that the although hygimar company manufactures

sanitary composite material from silica and unsaturated polyester orthophtalique (UPR). The synthesis shématise follows [1]:

where m = 1 - 3n = 10 - 30

Fig. 1. Structure of a typical polyester resin prepared from propylene glycol, maleic anhydride and phthalic anhydride and cross-linked with styrene.

According B.Bouss and other authors [5], the effect of temperature is negligible at 30° C, can be important to 40° C, and an extraordinary effect at 50° C.

Cutting chains begins at 200 ° C, and more thermal shock generates internal stresses which may lead to cracking of the resin. According to its authors, the oxidation temperature is maleic anhydride to 200 °C, and more carbonaceous chains are likely to degrade even at low amounts of oxygen.

Fig 2: Formation of the hydro peroxides groups falling on the thermo-oxidation of the resin. [6]



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Fig.3 Thermo-oxidation mechanism of the resin.[6]

Fig.4-Thermal decomposition mechanism of the resin. [6]

II. DEVELOPMENT AND CHARACTERIZATION OF NEW COMPOSITE

The material developed in this work is the unsaturated ortho-phthalic porcelain powder polyester containing increasing load rate of 25 to 100g introduced in UPR resin, other mixtures were made from silica and resin alone (sir) and mixtures containing different rates of porcelain powder, silica and resin (PPSiR) have been developed to compare the thermal behavior of our basic mixing and select the most appropriate formulation (Table. 1).. This resin is particularly adapted to the preparation of the thermosetting composite by its kind. This liquid is a resin which contains styrene and bidimentionnels chains. In the presence of a catalytic system consisting of benzoyl peroxide (diacyl peroxides) and cobalt salt, the resulting reaction between the two using equation (1) [4], there are cross-linking by formation of bridges and the cured resin.

ROOH +
$$Co^{2+}$$
 \rightarrow RO $^{+}OH^{-}$ + Co^{3+}
ROOH + Co^{3+} \rightarrow ROO $^{+}H^{+}$ + Co^{2+} (1)

Heat treatment of the porcelain powder is used in an oven at 110°C for 5 hours which is to reduce its water content to a value of zero or almost zero, this powder is size 180 micrometer.

The resin is characterized by a viscosity of 8 poises; it contains almost 34 % of styrene, density 1.13, an acid value of 25-30 mg KOH/g.

The reactivity characteristics are a gel time (4-6 min), curing (8-15 min) and the exothermic peak at $190 \,^{\circ}$ C.

Our composite was crafted in specimens. These specimens were exposed to varying temperatures from 25 to 220 °C.

Table.1.Percentages of specimens implemented from combinations of the matrix R, the silica (Si) and the porcelain powder (PP).

	PP1	PP2	PP3	PP4	Si1	Si2	
	R4	R4	R4	R4	R4	R4	
PP(g)	25	50	75	100	0	0	
Si(g)	0	0	0	0	25	50	
R(g)	100	100	100	100	100	100	
		Si3	Si4	PP1	PP2	PP3Si	
		R4	R4	Si3	Si2	1	
				R4	R4	R4	
PP(g)		0	0	25	50	75	
Si(g)		100	75	75	50	25	
R(g)		100	100	100	100	100	

III. RESULTS AND DISCUSSIONS

A. Relative humidity

weighed using a balance of precione startius 0.0001 an amount of the load, it is put into an oven at $110\,^{\circ}$ C for 24 hours, after we take the new mass and weighed.

This rate is calculated by the following equation Eq.(2).

$$^{\circ}/^{\circ}H = \frac{M_h - M_s}{M_h} * 100$$

with M_h is the weight of the moist material (g) and M_s is the mass of dry matter (g).

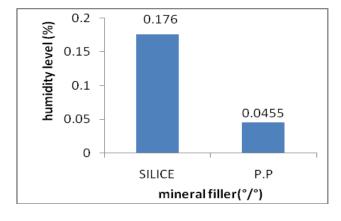


Fig. 5: relative humidity of two materials



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Figure (5) shows that the moisture content of the porcelain powder is almost zero which are an advantage for the development of our test specimens for the water elimination modified the surface of fillers; such subsequent penetration of the polyester resin in this charge was facilities. The particularity of this charge is a baked at 1300°C characterized by a near zero humidity, very high rigidity and a sufficiently large compact area.

B. Behavior of specimens with temperature

1. The weight loss

Weight loss (Δm %) on the ortho UPR resin (Figure.6) increases linearly with temperature and it is due to progressive evaporation of water_and the products, the loss reached 0.4 % to 50 ° C, not more than 3 °/° at 205 ° C and takes a maximum at 220 ° C (8 °/° C), this resin is thermally stable up to 125 °C.

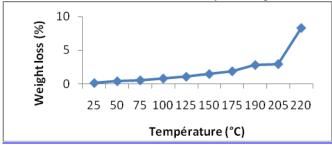


Fig.6. Evolution of the weight loss of the resin as a function of temperature

The weight loss for the PP / Ortho UPR combination (Figure 7) is an increasing function with temperature and decreases with the weight loading rate and therefore the thickness.

In Figure 7, we can see that for PP1R4, weight loss takes the values 1.7, 3.2 and 9 % successively to 175, 205 temperature to 220°C. While for PP2R4 combination weight loss takes successively 1.7,2.02 and $7.6^{\circ}/^{\circ}$ parallel temperatures 190,205 and 220°C but the material or weight percentages are equal weight loss is lower and reaches to values of 1.5 and 6 ° / 205 ° and 220°C.

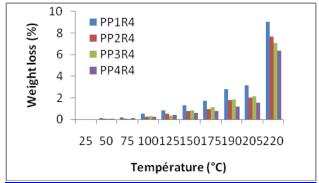


Fig. 7: Evolution of the loss in weight of the combination (PP / R) as a function of temperature.

The results are similar to the results obtained with the combination of silica/orthophthalic UPR (fig.8) that we have

to make a study on the specific combination of filler consisting of silica and the porcelain powder with the polymer matrix (fig. 9).

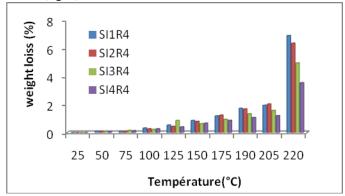


Fig.8 Evolution of the weight loss of the combination (Si / R) as a function of temperature

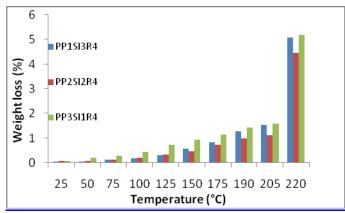


Fig.9 Evolution of the loss of weight of the combination (Si / PP / R) as a function of temperature

Taking these results on consideration, we can conclude that the loss of weight depends on two parameters which are the temperature and the weight percentage of the filler, the loss is caused by the water evaporation and the progressive dehydration of certain volatile organic compounds, then the loss reaches values of 1.5 for the combination PP4R4, 1.2 for combination of Si4R4 and 1.1 for that of PP2Si2R4. This parameter varies in the same way when the temperature varies from 25 to 220°C.

2. Damage to the specimens.

This loss of weight to the high temperatures from 150°C to 220 °C causes damage to processed products, this has been found by changing the color and appearance of cracks which are summarized in table 1.

The tracked changes show that these products have good thermal behavior to a temperature of $150^{\circ}C$ (no change was detected), according to some authors[2], the heat resistance of $130^{\circ}C$ reached polyester over $150^{\circ}C$, there is the beginning of the appearance of the crack path whose width is less than 0.2mm, the latter open linearly to form cracks between 0.2 and 2mm width, these cracks are active during the time based on thermal gradients and come in various forms (blind



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-through , surface crazing) [7] accompanies by a change in color and potentiality this parameter increases sulfide and measurement with the temperature , the appearance of these micro-cracks is a sign of degradation of samples prepared. During the temperature increase, it is a crack in the test specimen surface, whose thickness increases with the temperature to a greater cracking and increased, and in addition the position of the potential cracking that varies little with temperature and the nature of the combination.

Table.1- Changes with temperature of test specimens.

<u>test</u> specimens	Température (°C)										
	150	150	175	175	190	190	205	205	220	220	
	C	F	С	F	F	F	C	F	С	F	
PP1R4	N	Trace	M	##	M	##	M	##	M+N	##	
PP2R4	N	Trace	M	+	М	+	М	++	M+N	++	
PP3R4	N	Trace	M	+	М	++	М	#	M+N	+	
PP4R4	N	Trace	L	÷	M	+	M	+	M+N	+	
Si1R4	N	Trace	L	Traces	L	Traces	L	Traces+	M++	+++	
Si2R4	N	Traces	L	Traces		Traces		Traces+	M+	+++	
Si3R4	N	Trace	L	Traces		Traces		Traces+	M+	+	
Si4R4	N	Trace	L	Traces		Traces		Traces+	M+	++	
PP1Si3R4	N	Trace	L	Traces		Traces		Traces+	L	+	
PP2Si2R4	N	Trace	L	Traces		Traces		Traces+	L	+	
PP3Si1R4	N	Trace	M	Traces		Traces		Traces+	N	++	
R	M	+	M	+++	M	+++	M	++	M	++++	

C: color; F: Crack; M: brown; N: black; No change: -; L: light; + + + +: Important; + +: Medium; +: Weak.

According to the authors K. Laoubi [2] and others, the thermal degradation of the resin and the composite is produced in two steps: the first between 130 and 200°C and the second between 250 and 440°C, and more moderate temperatures (100°C), FT-IR analysis showed no thermal degradation of the resin. This result is in agreement with that obtained or composite resin and did not undergo any change up to 150°C.

Indeed, the damage specimens shall consist of a combination of cracking and oxidation of the matrix and defects in the volume of the material. The origin of the cracking is assigned to several factors at once:

The fragility of the matrix and the filler weakening binder/matrix power during the oxidation, the presence of reactive chemical species can also be the source of the first material degradation impurities such as additives, the constraints thermo-mechanical caused by differential expansion of the polyester resin and the porcelain powder, the degradation reactions of the scale such that the molecular macromolecular reconfigurations or displacements atoms.

IV. CONCLUSION

This study demonstrates the feasibility of developing composite from the porcelain powder. The new composite material is adopted good proprieties heat up to 150 °C, a homogeneous appearance which means that this new composite is well suited for applications sanitaire. Te filler content dispersed in the matrix has a binding power with the optimal matrix at a rate of 50 %, these results are satisfactory for Confirmed stiffness of this material, its tensile strength and its thermal behavior. The new material is thermally stable up to 100°C, which means the possibility of use as a sanitary product (sanitary product generally service temperature do not exceed 60°C).

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