

causes increasing thermal conductivity due to reduction the aerial pockets inside the insulating substance and the particle vibration conduct to heat and free electrons. Indeed despotic addition of the insulating substances depends on the media which have to be wanted of its resistance improvement was in contact with it, where add as fillers to lowering of thermal conduction at relatively high temperatures⁽²⁾. Where the substances are exposure to the direct flame heating, the fillers becomes a useless, therefore replaces by coating of the insulating substances have ability to endurance high temperature (some the cases more than 3000°C) which produced from the flame, then this materials are called a refractory, such as alumina oxide (Alumina Al₂O₃), silicon oxide (silica SiO₂), calcium oxide (Calicia CaO), and magnesium oxide (Magnesia MgO)⁽³⁾.

The aim of the present work is to study effect of heat treatment on the elected oxides for their use as insulators through improvement of water absorptivity and role of matrix substance

type through composite material carried on its.

Experimental work

Some oxides were used to preparation and manufacturing. the insulating substance, such as Aluminum oxide (Al₂O₃), calcium oxide (CaO), silicon oxide (SiO₂) and the calcium carbonate (CaCO₃) addition to the composite materials (unsaturated poly ester resin with random glassy fibers) and (resin with random glassy fibers). Apparent density was measuring for powders of insulating substances by weight of the empty bottleful (w₁) then weight it when its full with powder (w₂), puts ground in bottle measuring of the density and which known volume (V_c) (density cup) where the density inquired surface according to⁽⁸⁾:

$$\rho_A = \frac{w_2 - w_1}{V_c} \dots\dots(1)$$

Table (1) described the values of particle size, bulk and apparent densities for insulating powders. The tests were carried out in the Ibn Sinaa company.

Table (1) Physical properties for ceramic materials^(8,6).

compound	Al ₂ O ₃	CaO	SiO ₂	CaCO ₃
apparent density (g/cm ³)	0.8981	0.841	1.2146	0.6363
bulk density (g/cm ³)	1.5033	1.4749	1.4754	1.4868
particle size (µm)	8.38	11.16	13.16	6.53
Purity%	98.8	97	98.34	99.6

Preparing of matrix substance (the adhesive substance) and reinforcement fibers be consist of unsaturated polyester resin from Palatal A420 and epoxy LEyCO-POX 103 with specifications described in Table (2).

Table (2). Properties of epoxy resin ⁽¹¹⁾

Property	value
mixing ratio (%)	A:B=7:3
density at 23°C (g/cm ³)	1.05
min. curing temp. (°C)	10
Pot life 23°C-40°C (min)	40
overcoat min/max (23°C) (hr)	0-12
fully cured (23° C) (day)	7
linear shrinkage (%)	0.3
volume shrinkage (%)	3.5
compression strength (N/mm ²)	85-100
modulus of elasticity	2800
viscosity (N.sec/m ²)	75-91

Glassy fibers are used on form of random mat type E-glass, the physical properties of glassy fiber are described in Table (3).

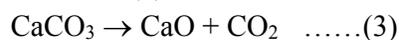
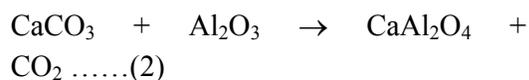
Table (3) physical properties of glassy fiber(10)

Property	value
density, g/cm ³	2.58
reactive Index	1.558
annealing point, °C	657
strain point, °C	615
tensile strength, Mpa at 25°C	3445
elongation %	4.8

Preparation of Specimens

The oxides are mixed with appropriate ratios using electrical stirrer for three hours for guarantee of the homogeneous distribution between the substances. Table (4) describe the mass ratios of test

specimens according to the stoichiometric reactions as follow :

**Table (4) Mass ratios of tested specimens.**

oxide	CaCO ₃ %	SiO ₂ %	CaO%	Al ₂ O ₃ %
sample 1				100
sample 2		65		35
sample 3	49			51
sample 4			36	64
sample 5	100			

The specimens were consist of two layers, the first layer is insulating layer from metal oxide or mixed of two oxides with appropriate ratios as in Table 4 , the thickness of this layer is 2 mm , the second layer consist of composite material from unsaturated polyester resin

or epoxy resin reinforced by random glassy fiber with 4 mm thick, as described in Fig. (1). An another type of specimen was manufactured from the composite layer only and called a blank to determine the absorptivity of the insulator layer.

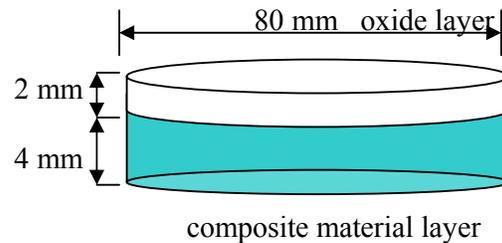


Fig. (1) diagram of specimen test

Water absorption test

The water absorptivity defined as the percentage of water mass passes through the porous of the solid to the weight of solid . The absorptivity is a function to the total porosity for the system which depends on the raw materials included in the material structure (type and structure of the substance), order of structure ,the particle size. The absorptivity test was carried out on two stages:

a. Before heat treatment

After coverage of the substance by powder of insulating materials where was use (10g) from the oxides or their mixture as in Table (1) . the absorption test carried on two types of specimen, the specimen carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers. In addition the third specimen is designed free from powder oxide. The absorptivity test carried out according to following steps:

1. weight the dry samples and registration of the reading w_1

2. immerse of the specimens in the distilled water at room temperature and for different time periods.
3. deduced the specimens from the from the distilled water after all lapse of time and drying , registration the weight (w_2) .
4. calculate increase in the weight (Δw_1) for blank specimen.
5. calculate increasing in the weight (Δw_2) for blank specimen coverage by powder of oxide material.
6. calculate the increasing in weight between step 4 and 5 which represent the absorbent water by oxide material.

$$\Delta w = \Delta w_2 - \Delta w_1$$

7. calculate the absorbtivity according to

$$Absorbitivity\% = \frac{w_2 - w_1}{w_1} \times 100$$

.... (7)

b. After heat treatment

1. record weight of the specimens before entering to the furnace.
2. Entering the specimens which carried on unsaturated polyester resin reinforced by glassy fibers at 150°C and for a period of 20 hour in

addition to the blank specimen to furnace, then entrance specimen of epoxy resin reinforced by glassy fibers at 125°C and for a period of 20 hour.

3. deduced the specimens from the furnace and left its to be cold then weight itss and registration (w_1).
4. immerse of the specimens in the distilled water at room temperature and for different time periods.
5. deduced the specimens from the from the distilled water after all lapse of time and drying, registration the weight (w_2).
6. calculate increasing in the weight (Δw_1) for blank specimen.
- 7 calculate increasing in the weight (Δw_2) for blank specimen coverage by powder of oxide material.
8. calculate the increasing in weight between step 4 and 5 which represent the absorbent water by the oxide material as above

Results and Discussion

Figs. (2), (3), (4), (5), (6), (7), (8) and (9) show the absorptive test for different specimens which be used in the present work. The results in case where the specimens carried on epoxy, we see that the experimental values are less than the values from thermal chemistry of reaction but in case of unsaturated polyester the values are higher than the experimental values. The absorbtivity is an osmosis and absorption of water on grains boundaries of substance inside the porous lines, consequently its depends on shape and size of particles, density, adhesion with the matrix and the voids formed between the oxide and matrix substamnce, therefore see a high change in absorbtivity on heating of the samples for long periods under high temperatures,

where that the heat treatment have high incidence on all the parameters have mentioned above given a very high absorbtivity compared with results of the unheated specimens.

When two oxide are mixed with equal ratios hence the absorbtivity of new structure is not algebraic addition of the their constituents, this indicate that the physical properties of oxide are different for new mixture as described in Table (6). Fig.(5-9) show comparisons of the specimens before and after the heat treatment, notice that the difference in the absorbtivity values due to gaps and voids growth in the interior structure of the substance or mixtures, for the same reason the difference behavior between the specimens carried on epoxy resin and polyester resin.

Fig.(6) show the absorbtivity of ($Al_2O_3-SiO_2$) mixture, the difference is physically due to the heating and activation this mixture, consequently change of its arrangement as absorbent substances for water.

Fig.(10) show the absorbtivity test of specimens carried on epoxy rein befor heat treatment, notice increase the weight against time until settles at superior increase in the weight due to the specimens becomes saturation as described in Table (6). Calcium oxide gives a highest absorbtivity 9.6% because its caustic oxide interaction with the water when is the oxide as powder and absorbs the water when its compressed as solid layer, this result be reversed on mixture $CaO-Al_2O_3$ and mixture $CaO-SiO_2$ have absorbtivity 11.2% and 10.4% respectively. even though the absorbtivity results from the algebraic addition of absorbtivity values of constituent the mixture were the results becomes 7.9%

and 6% respectively, the increase returns to porosity number of voids in the mixture other than when solitary substance. The absorptivity of regimes above agrees with solitary substances where sees that the absorbtivity of the mix contains alumina is higher than that which contains silica in spite of the absorbtivity of silica is higher than alumina.

Fig. (11) described the absorptivity test for specimens carried on epoxy resin, so notice that when the specimens are heated for 20 hours at 125°C. The absorptivity was increased due to growth of the gaps and porous trough powder. Cohesion among powder particles and adhesion between particles of powder and matrix, the heating of oxide layer to this temperature performs a matrix softness and stripping the gasses present in the oxide. The maximum increases which occurred to absorptivity in regimes of CaCO_3 % 17.7, $\text{Al}_2\text{O}_3\text{-SiO}_2$ % 14.4, $\text{Al}_2\text{O}_3\text{-CaCO}_3$ and SiO_2 % 11.3.

Fig. (12) Show absorptivity test of specimens carried on unsaturated polyester resin, so notice that as in Table (6), absorptivity for the same mixtures above higher than of the specimens carried on epoxy resin due to adhesion forces between oxide and matrix layers, consequently the interface included avoids and porous which allow to absorption of

water other than the species carried on epoxy resin.

Fig. (13) represent absorptivity test for specimens carried on unsaturated polyester resin with heat treatment, so notice that where the specimens are heated to 150°C for a period 20 hour, the heat treatment is sufficient to regenerate this materials physically to strip the adsorption gasses through the interface layer to produce a more stable regimes which have a high efficiency to absorption of water, then gave a good absorptivity have a multiple values result from epoxy resin. That reverses special all substance during the activation operation and disagreement of each material behavior physically under conditions of the activation above so the values are differet from unheated of substances, consequently change of their arrangement as absorbent substances for water.

Table(5) maximum weight of water absorption.

Material (10g)	Δw_{\max} (g) water absorbed by the material on Epoxy matrix		Δw_{\max} (g) water absorbed by the material on unsat polyester matrix	
	Un heated	Heated 125°C	Unheated	Heated 150°C
Al ₂ O ₃	0.63	0.9	1.7	3.9
CaO	0.96	1.04	3.6	10.8
SiO ₂	0.25	1.13	1.5	2.3
CaCO ₃	0.2	1.77	1.7	2.4
Al ₂ O ₃ -CaO	1.12	1.2	1.2	4.6
Al ₂ O ₃ -SiO ₂	0.1	1.44	1.1	1.7
Al ₂ O ₃ -CaCO ₃	0.12	1.4	2.2	2.8
CaO-SiO ₂	1.04	1.08	6	9.3

Table (6) Absorptivity of oxides and their mixtures.

Material (5g/5g)	%Absorptivity by the material on Epoxy matrix		%Absorptivity by the material on unsat. Polyester matrix	
	Un heated	heated to 125°C	Unheated	Heated to 150°C
Al ₂ O ₃	6.3	9	17	39
CaO	9.6	10.4	36	108
SiO ₂	2.5	11.3	15	23
CaCO ₃	2	17.7	17	24
Al ₂ O ₃ -CaO	11.2	12	12	46
Al ₂ O ₃ -SiO ₂	1	14.4	11	17
Al ₂ O ₃ -CaCO ₃	1.2	14	22	28
CaO-SiO ₂	10.4	10.8	60	93

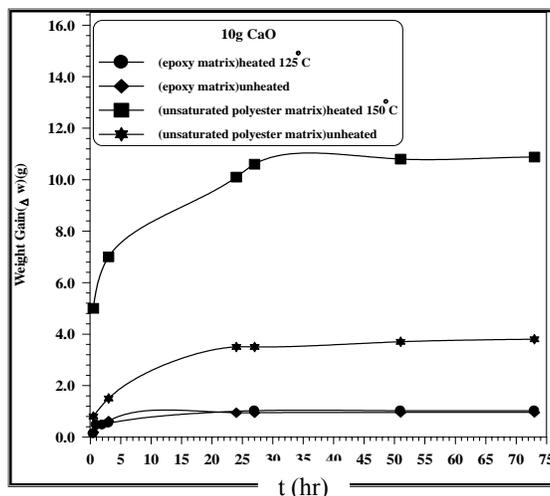


Fig.(2) Aabsorptivity test for CaO carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers .

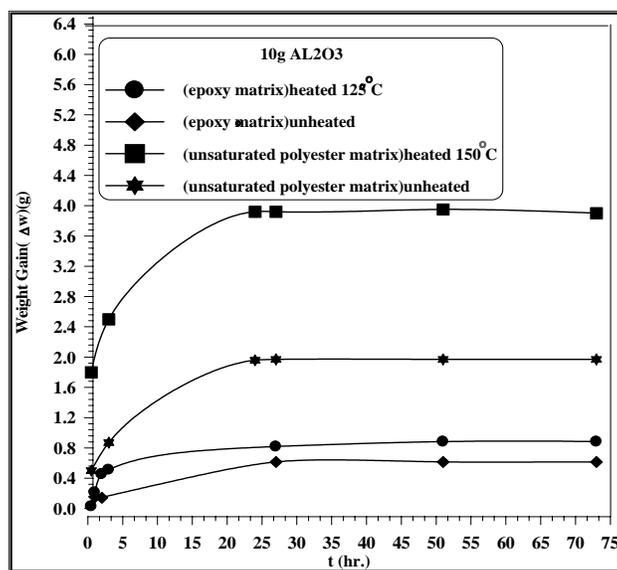


Fig.(3) Aabsorptivity test for Al₂O₃ carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers .

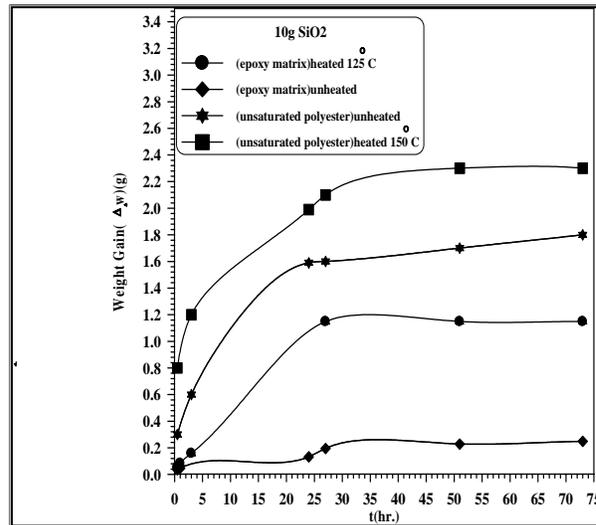


Fig.(4) Absorptivity test for SiO₂ carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers .

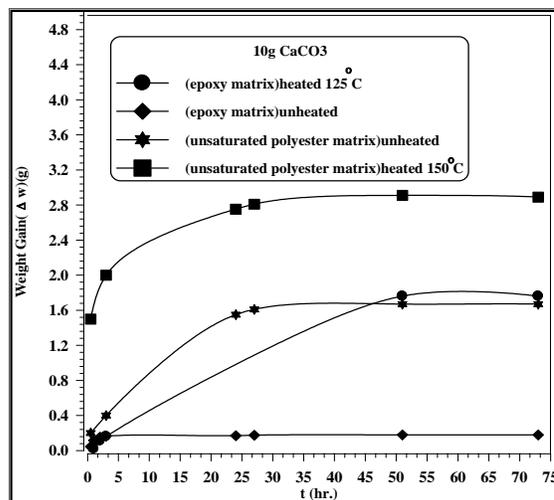


Fig.(5) Absorptivity test for CaCO₃ carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers .

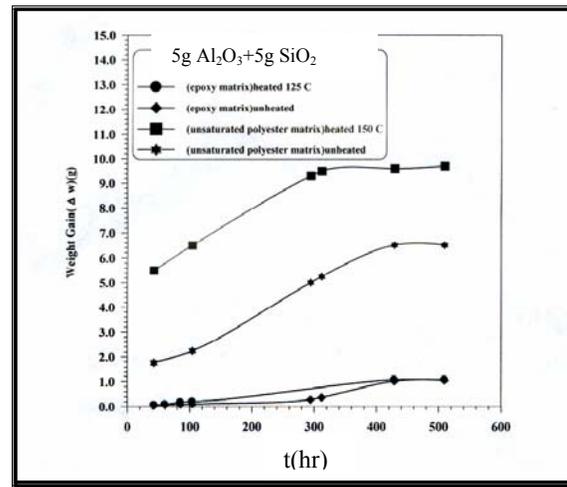


Fig.(6) Absorptivity test for Al₂O₃- SiO₂ mixture carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers .

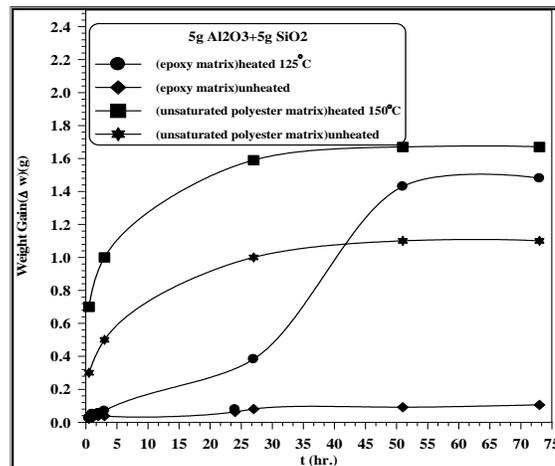


Fig.(7) Absorptivity test for system of Al₂O₃- CaO mixture carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers.

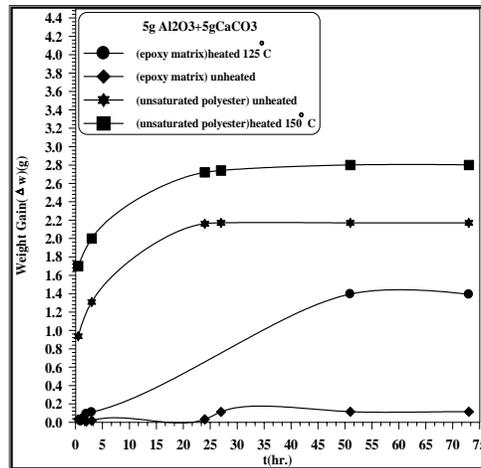


Fig.(8) Aabsorptivity test for system of Al₂O₃- CaCO₃ mixture carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers

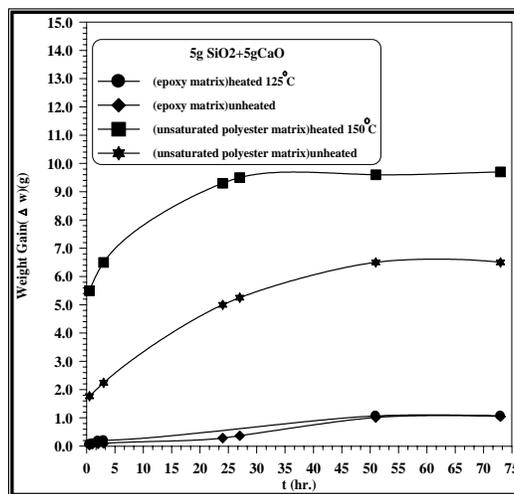


Fig.(9) Aabsorptivity test for system of SiO₂- CaO mixture carried on unsaturated polyester resin reinforced by glassy fibers and other carried on epoxy resin reinforced by glassy fibers.

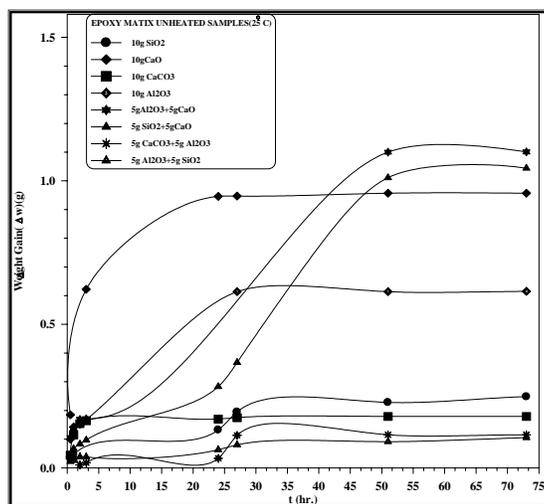


Fig.(10) Absorptivity test for species carried on epoxy resin reinforced by glassy fibers at 25 °C .

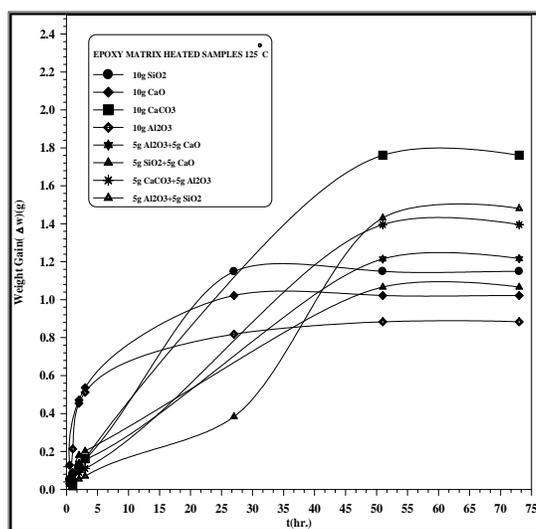


Fig.(11) Absorptivity test for species carried on epoxy resin reinforced by glassy fibers heated to 125 °C.

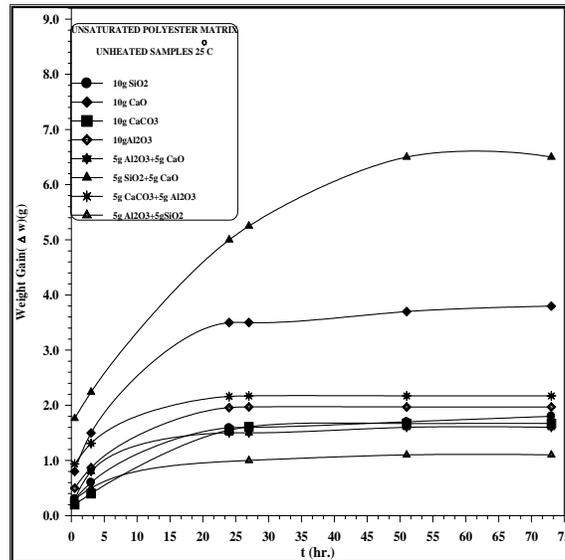


Fig.(12) Absorptivity test for species carried on unsaturated polyester resin reinforced by glassy fibers at 25 °C

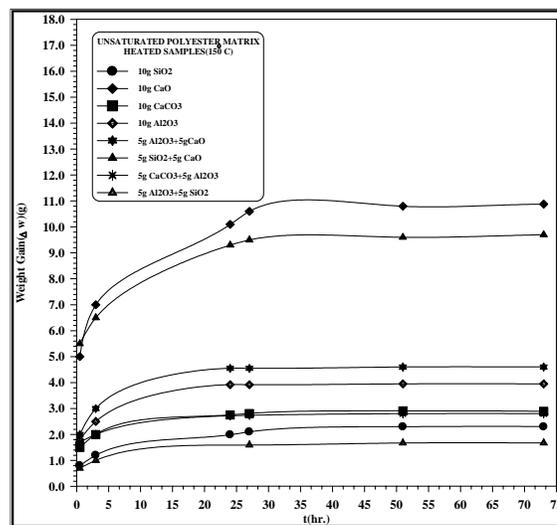


Fig.(13) Absorptivity test of species carried on unsaturated polyester resin reinforced by glassy fibers heated to 150 °C

Conclusion

By examining water absorptivity of oxides as a function of time at constant temperature. The results show that the absorptivity of the oxides varies as:

1. The absorptivity of samples carried on epoxy matrix without heat treatment, followed the order $\text{Al}_2\text{O}_3\text{-CaO} > \text{CaO-SiO}_2 > \text{CaO} >$

- Al₂O₃ > SiO₂ > CaCO₃ > Al₂O₃-CaCO₃ > Al₂O₃-SiO₂ .
- The absorbitivity of samples carried on epoxy matrix with heat treatment at 125 °C for a long period, followed the order CaCO₃ > Al₂O₃-SiO₂ > Al₂O₃-CaCO₃ > Al₂O₃-CaO > SiO₂ > CaO-SiO₂ > CaO > Al₂O₃ .
 - The absorbitivity of samples carried on unsaturated polyester matrix without heat treatment, followed the order CaO-SiO₂ > CaO > Al₂O₃ - CaCO₃ > Al₂O₃ - CaCO₃ > SiO₂ > Al₂O₃- CaO > Al₂O₃-SiO₂ .
 - The absorbitivity of samples carried on unsaturated polyester matrix with heat treatment at 150 oC for a long period, followed the order CaO > CaO-SiO₂ > Al₂O₃-CaO > Al₂O₃ > Al₂O₃-CaCO₃ > CaCO₃ > SiO₂ > Al₂O₃- SiO₂ .
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