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## Preparation and performance evaluation of jute and glass composites of epoxy resin of (2E,6E)-2,6-bis(4-hydroxy benzylidene)-4-methylcyclohexanone

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### ABSTRACT

Jute and glass composites were prepared by compressing molding technique at 150°C for 3h under 20 MPa pressure. J-EMHBC-DDS, J-EMHBC-THPA, G-EMHBC-DDS and G-EMHBC-THPA showed 15, 15.1, 58.3 and 18.7 MPa tensile strength, respectively. J-EMHBC-DDS and J-EMHBC-THPA showed 22.9 and 19.4 MPa flexural strength, respectively. J-EMHBC-DDM and G-EMHBC-DDM showed  $1.6 \times 10^9$  and  $1.2 \times 10^{12}$  ohm cm volume resistivity, respectively. J-EMHBC-DDM and G-EMHBC-DDM showed 2.8 and 11.2 kVmm<sup>-1</sup>, respectively. Water uptake in the composites was carried out in pure water and 10% each of aqueous HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, NaOH, KOH and NaCl solutions at 35 °C as well as in boiling water. Composites showed high water uptake tendency and longer equilibrium time in different environments. High water uptake in the composites is due to presence of hydroxyl groups in celluloses and resins and also due to formation of micro cracks. Nature of the electrolytes also played an important role on water uptake behavior and diffusivity in the composites. Drastic reduction in equilibrium water uptake time was observed in boiling water.

**Keywords:** Photosensitive epoxy resin, Composites, Tensile strength, Flexural strength, Electric strength, Volume resistivity, Water uptake, Diffusivity

## **1. INTRODUCTION**

Epoxy resins are thermosetting polymers that, before curing, have one or more active epoxide or oxirane group(s) at the end(s) of the molecule and a few repeated units in the middle of the molecule<sup>1,2</sup>. They exist either as liquids with lower viscosity or as solids and therefore their molecular weights can vary greatly. Epoxy resins are extensively investigated for high performance industrial and engineering applications. They are most widely employed in the coatings, adhesives, matrices for advance polymer composites, joining and fastening technology, electronic and electrical equipments, low-stress IC encapsulants because of their great versatility and characteristic very good to excellent thermo-mechanical, electrical and chemical resistance properties and low shrinkage on cure<sup>3-10</sup>, excellent adherence to many substrates<sup>11,12</sup> but their applications are often restricted due to their poor toughness. Hyper branched polyesters<sup>13,14</sup> can be used as tougheners to improve the toughness of epoxy resins because of their high density of functional terminal groups and their low viscosity make them possible to improve processability of the epoxy resins. For advanced microelectronic and packaging technology, the epoxy resins must possess improved good thermal stability, low dielectric constant and dissipation factor, low water absorption, low coefficient of thermal expansion, low internal stress, high mechanical strength, and low modulus<sup>15-18</sup>.

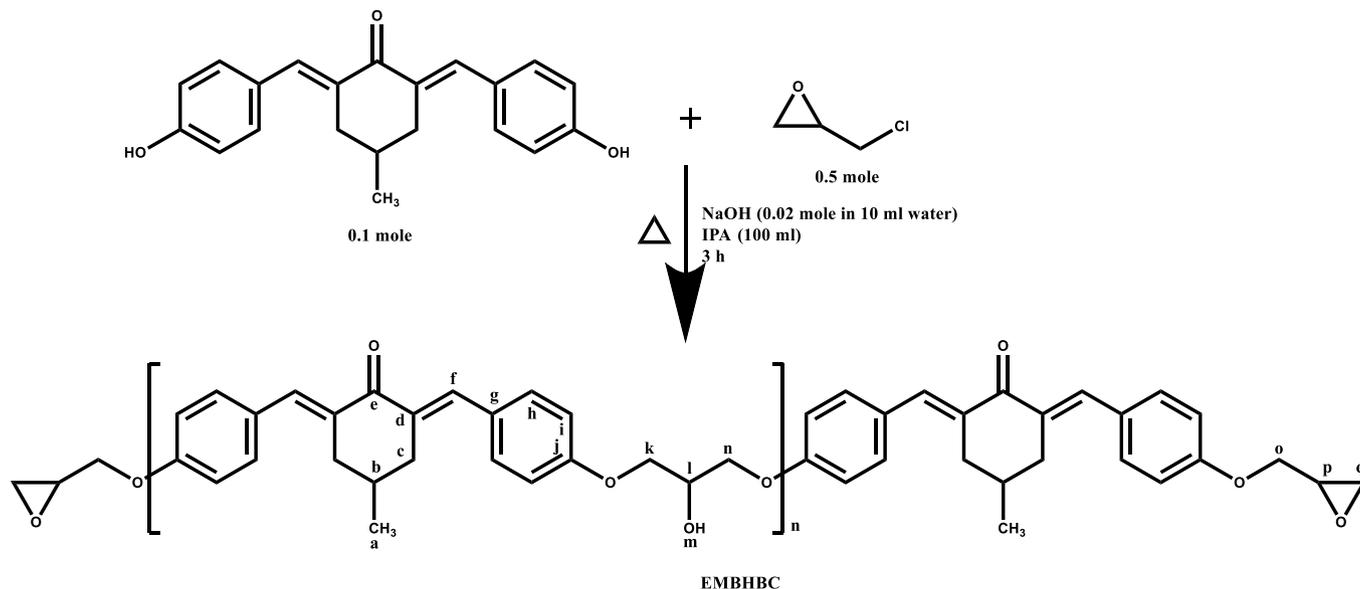
To the best of our knowledge, limited literature is available on epoxy resins containing chalcone moiety as photosensitive materials<sup>9,18,19</sup>, which prompted us to undertake present piece of work. In present work we have reported preparation of jute/glass composites of epoxy resin of (2E,6E)-2,6-bis(4-hydroxy benzylidene)-4-methylcyclohexanone and their performance evaluation.

## **2. EXPERIMENTAL**

### **2. 1. Materials and methods**

Solvents and chemicals used in this study were of LR grade and were used as received or purified according to reported methods<sup>20</sup>. Woven silane treated E-glass fabric used in the present study was collected from the Unnati Chemicals, Ahemdabad and jute fabric (brown jute) was purchased from local market, Rajkot. Mylar film and silicone spray were used as mold releasing agents. The epoxy resin of (2E, 6E)-bis(4-hydroxy benzylidene)-4-methylcyclohexanone (EMBHBC) was synthesized and purified according to our recent communication (Scheme-I)<sup>19</sup>. The epoxy equivalent weight of the resin was 898.3. 4,4'-Bis(4-aminophenyl)methane (DDM), 4,4'-bis(4-aminophenyl)sulphone (DDS) were supplied by National Chemicals Vadodara and tetrahydrophthalic anhydride (THPA) was supplied by Merck, Germany. The hardeners were used as received. Chloroform was supplied by Allied Chemical Corporation, Vadodara. The tensile strength (IS: 11,298-Pt-2-87) and flexural strength (ASTM-D-790-92), electric strength (ASTM-D-149-2009), and volume resistivity (ASTM-D-257-2014) measurements were made on a Universal tensile testing machine (Model No. 1185) at a speed of 50 mm/ min, a high voltage tester (Automatic Electric, Mumbai) in air at 27 °C by using 25/75-mm brass electrodes, and a Hewlett-Packard high resistance meter at 500 V DC after charging for 60 s, respectively. Water absorption study was carried out at 35 °C by using water and 10% aq. each of HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, NaCl, NaOH and KOH and also in boiling water by change in mass method. Each of the composites samples were weighed by

using Contech electronic balance and placed in stoppered flasks containing water, acid, alkali and salt solutions at 35 °C with intermittent stirring. After 24 h, the samples were taken out, dried with tissue papers, reweighed and reimmersed in respective solutions. The measurements were carried out till constant weights were observed. Similarly water absorption study in boiling water was carried out at the interval of 1 h.



**Scheme I**

## 2. 2. Fabrication of Jute/Glass Composites of Epoxy Resin

Jute and glass fibers reinforced composites of 20 cm × 20 cm of EMBHBC were prepared by a hand layup compression molding technique using stoichiometric amount of 4,4'-bis(4-amino phenyl)methane (DDM), 4,4'-bis(4-amino phenyl)sulphone (DDS) and 1,2,3,6-tetrahydro phthalic anhydride (THPA) as hardeners. Equal weights of EMBHBC and reinforcing fibers were taken for the preparation of the composites. The required quantities of EMBHBC and DDM / DDS / TPHA (Table 1) were dissolved in 170 ml chloroform. The resultant solution was heated at 50 °C with stirring for 10 min and cooled to room temperature. The resultant solution was applied to jute/glass fabrics with the help of a smooth brush and prepregs were allowed to dry in the sunlight for about 15 min. Ten such prepregs of woven glass and eight prepregs of jute were stacked one over the other and pressed between two preheated stainless steel plates under the hydraulic pressure of 20 MPa and at 150 °C for 3 h and 4 h at room temperature. Silicone spray was used as a mold releasing agent. Here after the composites are designated as J-EMBHBC-DDM, J-EMBHBC-DDS and J-EMBHBC-THPA for jute composites; and G-EMBHBC-DDM, G-EMBHBC-DDS and G-EMBHBC-THPA for woven glass composites. For tensile and flexural strength measurements, the samples were machined according to standard test methods. For water uptake study in various environments, 2 cm × 2 cm samples were cut and edges were sealed with a respective EMBHBC-DDM / DDS / TPHA solution and cured at their respective curing temperature for 3 h.

Measurements

**Table 1.** Experimental details for the preparations of jute/ glass composites.

Composite	EMBHBC, g	Fiber, g	Amount of hardener, g	Temperature, °C	Time, h
J-EMBHBC-DDS	147.10	Jute	20.33	150	3
J-EMBHBC-THPA	156.83	Jute	26.54	150	3
J-EMBHBC-DDM	51.74	Jute	5.71	150	3
G-EMBHBC-DDS	132.23	Glass	18.27	150	3
G-EMBHBC-THPA	130.03	Glass	22.05	150	3
G-EMBHBC-DDM	30.32	Glass	3.35	150	3

### 3. RESULTS AND DISCUSSION

#### 3. 1. Mechanical properties

Tensile properties of materials are beneficial for engineering design and understanding quality features of polymeric materials. Flexural properties are useful for quality control and categorization of materials with respect to stiffness and bending strength. Flexural strength is the resistance of the material under the bending mode. The tensile strength ( $\sigma$ ) of the composites was determined according to Eqn. 1:

$$\sigma = \frac{W}{A} \quad (1)$$

where  $W$  is the load value at any point on the load extension curve and  $A$  is the cross sectional area of the sample. J-EMBHBC-DDS, J-EMBHBC-THPA, G-EMBHBC-DDS and G-EMBHBC-THPA showed 15, 15.1, 58.3 and 18.7 MPa tensile strength, respectively.

Flexural strength is the resistance of the material under the bending mode. The flexural strength of the composites was determined according to Eqn. 2:

$$\text{Flexural strength} = \frac{1.5 FL}{wt^2} \quad (2)$$

where  $F$  is the breaking load,  $L$  is the span length,  $w$  is the width and  $t$  is the thickness of the specimen. J-EMBHBC-DDS and J-EMBHBC-THPA showed 22.9 and 19.4 MPa flexural strength, respectively. J-EMBHBC-DDS and J-EMBHBC-THPA showed improved flexural property.

Jute-epoxy and glass-epoxy composites showed moderate tensile and flexural properties due to poor interfacial adhesion. It was observed that during processing and machining composites showed delaminating tendency in some cases indicating hard and brittle nature of the cured resin. It is expected that increasing fiber loading increases strength, which means effective stress transfer between reinforcement and the matrix. As compared to J-ECH-TEA

(96.5 MPa tensile strength and 84 MPa flexural strength) and G-ECH-TEA (275 MPa tensile strength and 351 MPa flexural strength)<sup>9</sup>. The composites under investigation showed much inferior studied mechanical properties.

Jute fibers are mainly made up of cellulose, hemicellulose and lignin. Jute fibers are hygroscopic in nature and possess poor wettability with hydrophobic resins and as a result poor fiber matrix interfacial bond strength is expected<sup>21,22</sup>. The mechanical properties of natural fiber reinforced composites depend upon a number of parameters namely fiber strength and modulus, fiber length and orientation, fiber matrix interfacial bond strength, compatibilizer and impact modifier, and fiber content. Fiber-matrix interface plays an important role in the composite properties. A good interfacial bond is required for effective stress transfer from the matrix to the fiber, whereby maximum utilization of the fiber strength in the composite is achieved. Matrix interface adhesion and particle loading are two important factors that also affect mechanical properties. The use of coupling agents that also increase the matrix adhesion leads to higher strength.

Flexural strength of the composite is almost entirely provided by the reinforcement. Increase in the flexural strength indicated an increase in resistance to shearing, while decrease in the flexural strength is the evidence of the reduction in the stiffness of the composite. The performance and durability of the composites depend upon the strength and stiffness of the fibers, the strength and stability of matrix and the interfacial bond strength between fiber and matrix.

Oriented fibers are strong when pulled in the direction of the fiber but they are weak at right angles to the pulled direction. The woven fibers give a composite good strength in many directions. Flexural property of the composites depends upon various factors such as the type and amount of the additives, which can soften or reinforce the material, sample preparation technique, increasing temperature decreases strength and modulus, surface roughness, sinks, voids and any other kinds of imperfection, anisotropy, sample dimensions, etc.

### 3. 2. Electrical properties

Volume resistivity and electrical strength data are helpful for comparing relative insulation quality of material selection to analyze the effect of material composition and environment. Synthetic organic polymers are well known for their electrical insulation characteristic and offer a wide choice of mechanical properties in combinations ranging from flexible to very rigid and strong. Polymers can be tailored to obtain specific properties in combinations. Volume resistivity of the jute and glass composites was determined according to Eqn. 3:

$$\text{Volume resistivity} = R_v \frac{A}{t} \quad (3)$$

where  $R_v$  is the volume resistance in ohms,  $A$  is the area of electrodes and  $t$  is the thickness of the specimen in cm. J-EMBHBC-DDM and G-EMBHBC-DDM showed  $1.6 \times 10^9$  and  $1.2 \times 10^{12}$  ohm cm volume resistivity, respectively.

Electric strength of the jute and glass composites was determined according to Eqn. 4:

$$\text{Electric strength} = \frac{V}{t} \quad (4)$$

where  $V$  is the puncture voltage in volt and  $t$  is the thickness of the specimen in mm. J-EMBHBC-DDM and G-EMBHBC-DDM showed 2.8 and 11.2 kVmm<sup>-1</sup>, respectively. Both composites showed fairly good volume resistivity and electric strength indicating somewhat polar nature of the composites. As compared to J-ECH-TEA (1.1×10<sup>11</sup> ohm cm volume resistivity and 7.1 kVmm<sup>-1</sup> electric strength) and G-ECH-TEA (1.4×10<sup>13</sup> ohm cm volume resistivity and 24.6 kVmm<sup>-1</sup> electric strength)<sup>9</sup> J-EMBHBC-DDM showed much lower electrical properties.

The nature and structure of the hardeners have affected mechanical and electrical properties of the jute and glass composites. Electrical properties of the fiber reinforced composites depend upon various factors like temperature, nature of resin, humidity, degree of resin cure, electrode area and electrode material, sample thickness, time of voltage application, and current frequency, etc.

### 3. 3. Water uptake study

NFRCs are susceptible to moisture uptake because of hydrophilic hydroxyl groups of cellulose, hemicellulose, and lignin. Moreover, natural fibers possess poor wettability with hydrophobic resins<sup>9,22,23</sup>. Hemicellulose is mainly responsible for moisture uptake and responsible for the deterioration of mechanical properties. Wateruptake in polymeric composites is proved to be Fickian as well as non-Fickian in character<sup>24</sup>. The viscoelastic nature of polymeric materials and the formation of cracks during processing and during service are responsible for non-Fickian diffusion of water in composites.

Assuming unidirectional Fickian diffusion of water in composite, water uptake in J-EMBHBC-DDS, J-EMBHBC-THPA, G-EMBHBC-DDS and G-EMBHBC-THPA was carried out at a 35°C in various environments namely pure water and 10% each of aqueous HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, NaOH, KOH and NaCl solutions as well as in boiling water. Percent weight gain (M) during ageing in the composites with the passage of time was determined according to Eqn. 5:

$$M = \frac{W_m - W_d}{W_d} \times 100 \quad (5)$$

where,  $W_m$  and  $W_d$  are the weights of the moist and dry sample, respectively. Percent weight gain against the square root of soaking time curves for J-EMBHBC-DDS, J-EMBHBC-THPA, G-EMBHBC-DDS and G-EMBHBC-THPA in various environments at 35 °C are shown in Figs. 1-4, respectively from which it is observed that water uptake behavior in different environments is quite different indicating different diffusion behavior and hence Fickian like water uptake behavior. The percent weight gain increased, reached to maximum value and then leveled off, when the equilibrium was established. Equilibrium water uptake and the corresponding equilibrium time for each of the composites are reported in Table 2. Observed trends for the % equilibrium water uptake in the composites and corresponding equilibrium time are as follow:

For equilibrium water uptake

J-EMBHBC-DDS: NaOH > KOH > HCl > HNO<sub>3</sub> > NaCl > H<sub>2</sub>O > H<sub>2</sub>SO<sub>4</sub>

J-EMBHBC-THPA: NaOH > KOH > H<sub>2</sub>SO<sub>4</sub> > HCl > H<sub>2</sub>O > HNO<sub>3</sub> > NaCl

G-EMBHBC-DDS: H<sub>2</sub>SO<sub>4</sub> > KOH > H<sub>2</sub>O > HCl > NaCl > HNO<sub>3</sub> > NaOH

G-EMBHBC-THPA:  $H_2SO_4 > KOH > NaOH > H_2O > HCl > HNO_3 > NaCl$

For equilibrium time

J-EMBHBC-DDS:  $HNO_3 > NaCl > H_2SO_4 > NaOH > HCl > H_2O > KOH$

J-EMBHBC-THPA:  $NaCl > KOH > HCl > HNO_3 > H_2O > H_2SO_4 > NaOH$

G-EMBHBC-DDS:  $NaCl > H_2SO_4 > KOH > NaOH > HNO_3 > HCl > H_2O$

G-EMBHBC-THPA:  $HCl > KOH > NaOH > HNO_3 > H_2SO_4 > H_2O > NaCl$

**Table 2.** The equilibrium water content, diffusivity and the equilibrium time data of composites in different environments at 35 °C.

Environment	Thickness, mm	Equilibrium water content, %	Equilibrium time, h	Diffusivity $D_x \times 10^{12} \text{ m}^2\text{s}^{-1}$
<b>J-EMBHBC-DDS</b>				
HCl	4.65	20.1	350	7.65
HNO <sub>3</sub>	4.63	17.8	618	6.20
H <sub>2</sub> SO <sub>4</sub>	4.56	15.0	586	4.24
NaCl	4.61	16.8	598	5.19
NaOH	4.62	24.4	384	5.98
KOH	4.63	23.7	324	5.91
H <sub>2</sub> O	4.51	15.1	334	6.0
Boiling water	4.61	25.2	2	73.82
<b>J-EMBHBC-THPA</b>				
HCl	4.47	18.3	431	6.00
HNO <sub>3</sub>	4.47	13.2	408	5.70
H <sub>2</sub> SO <sub>4</sub>	4.43	25.9	284	21.92
NaCl	4.47	10.7	457	8.86
NaOH	4.47	82.2	118	15.55
KOH	4.46	52.8	438	7.80
H <sub>2</sub> O	4.45	17.4	385	5.46

Boiling water	4.47	24.2	4	150.32
G-EMBHBC-DDS				
HCl	1.79	14.8	506	0.59
HNO <sub>3</sub>	1.88	13.6	509	0.88
H <sub>2</sub> SO <sub>4</sub>	1.92	43.6	573	0.33
NaCl	1.96	13.8	616	0.09
NaOH	1.96	12.4	530	0.93
KOH	1.79	21.9	549	0.11
H <sub>2</sub> O	1.80	20.2	410	0.15
Boiling water	1.88	24.3	2	1.94
G-EMBHBC-THPA				
HCl	1.94	20.3	622	0.11
HNO <sub>3</sub>	1.97	10.9	483	0.92
H <sub>2</sub> SO <sub>4</sub>	1.89	62.9	455	0.98
NaCl	1.80	9.8	429	1.51
NaOH	1.97	28.5	528	0.68
KOH	1.85	35.9	530	0.66
H <sub>2</sub> O	2.03	20.5	433	0.44
Boiling water	1.89	36.4	2	50.92

High water uptake in the composites is mainly due to presence of hydrophilic hydroxyl groups in celluloses and epoxy resin and also due to solvated ether, amine / ester groups. In some cases very high water uptake in a specific environment is due to formation of micro cracks. The observed difference in equilibrium water absorption in the composites is due to different nature of hardeners and different curing behavior. Nature of electrolyte solutions also affected water uptake and diffusivity of solvated water in the composites. Strong electrolytes break water structure and hence the size of the solvated ions, which affected diffusivity in the composites. Water diffusion mechanism in the composites takes through water diffusion directly into the matrix and reach the fibers or they enter the composite by capillary action along the fiber-matrix interface followed by diffusion from the interface into the bulk resin. The rate of water diffusion depends on external environments such as temperature, applied stress, internal material states such as debonding at the fiber-matrix interface, voids, additives, fiber

loading, fiber arrangement, matrix cracking and the inherent sorption property of the constituent materials. Beyond saturation point water exists as free water in the void structure leading to composite delamination or void formation<sup>25</sup>. Absorbed water causes weakening of interfacial bonding and results in lowering of the mechanical strength. Formation of voids and blistering causes high water absorption<sup>26</sup>. It is well documented in the literature<sup>27,28</sup> that mechanical properties are degraded after water absorption ageing. Absorption of water in composites causes swelling of fibers till the cell walls are saturated with water and beyond that water exists as free water in the void structure leading to composite delamination or void formation<sup>29</sup>. Absorbed water causes weakening of interfacial adhesion and hence hydrolytic degradation of both matrix and fibers<sup>30,31</sup>. Cracking and blistering of fibers cause high water absorption, while degradation causes leaching of small molecules<sup>32-34</sup>.

### **3. 4. Diffusivity (D<sub>x</sub>)**

With the consideration that absorbed water plays a significant role in influencing mechanical behavior, especially tensile strength, long-term durability of the polymer, and polymer matrix composites, we have carried out water uptake behavior in the different chemical environments by assuming one-dimensional diffusion only. Diffusivity in the composites is related to water uptake with the passage of time according to Eqn. 6:

$$M = \frac{4M_m}{h} \sqrt{\frac{t}{\pi}} \sqrt{D_x} \quad (6)$$

where M<sub>m</sub> is the equilibrium water content in the sample, D<sub>x</sub> is the diffusivity and t is time.

Diffusivity in a given environment can be determined from the initial slope of the plot of M against the square root of time:

$$D_x = \pi \left( \frac{h}{4M_m} \right)^2 (\text{Slope})^2 \quad (7)$$

Diffusivities in different environments was determined according to eqns. 6 and 7 by determining initial slopes of M against t<sup>1/2</sup> curves and are reported in Table 2 from which it is observed that diffusivities in the different environments are different due to different nature of the electrolyte solutions depending upon different solvated ionic sizes. Thus, the size of the solvated ions also caused diffusion rate.

### **3. 5. Water Uptake in Boiling Water**

Water uptake in composites depends mainly on temperature besides the presence of hydrophilic groups in the composites. The effect of boiling water on percent water uptake with time is shown in Fig. 5 from which it is evident that saturation water uptake in all composites is observed after 5 h and then remained almost constant with time. The increase in temperature caused an increase in diffusion rate and drastic reduction in equilibrium water uptake time. The equilibrium water uptake is observed more than that of at 35 °C due to formation of micro cracks. Chemical changes occurred due mainly to the hydrolysis of polymer bonds, dissolution, and leaching of the water soluble species. Therefore, the extent of degradation of polymer matrix at elevated temperature is expected to be greater than that at a lower temperature.

#### 4. CONCLUSIONS

On the basis of the experimental findings, it is concluded that jute/glass-epoxy composites displayed fairly moderate mechanical and electrical properties. Composites showed high water uptake tendency and longer equilibrium time in different environments. Nature of the electrolytes also played an important role on water uptake behavior and diffusivity in the composites. Drastic reduction in equilibrium water uptake time was observed in boiling water.

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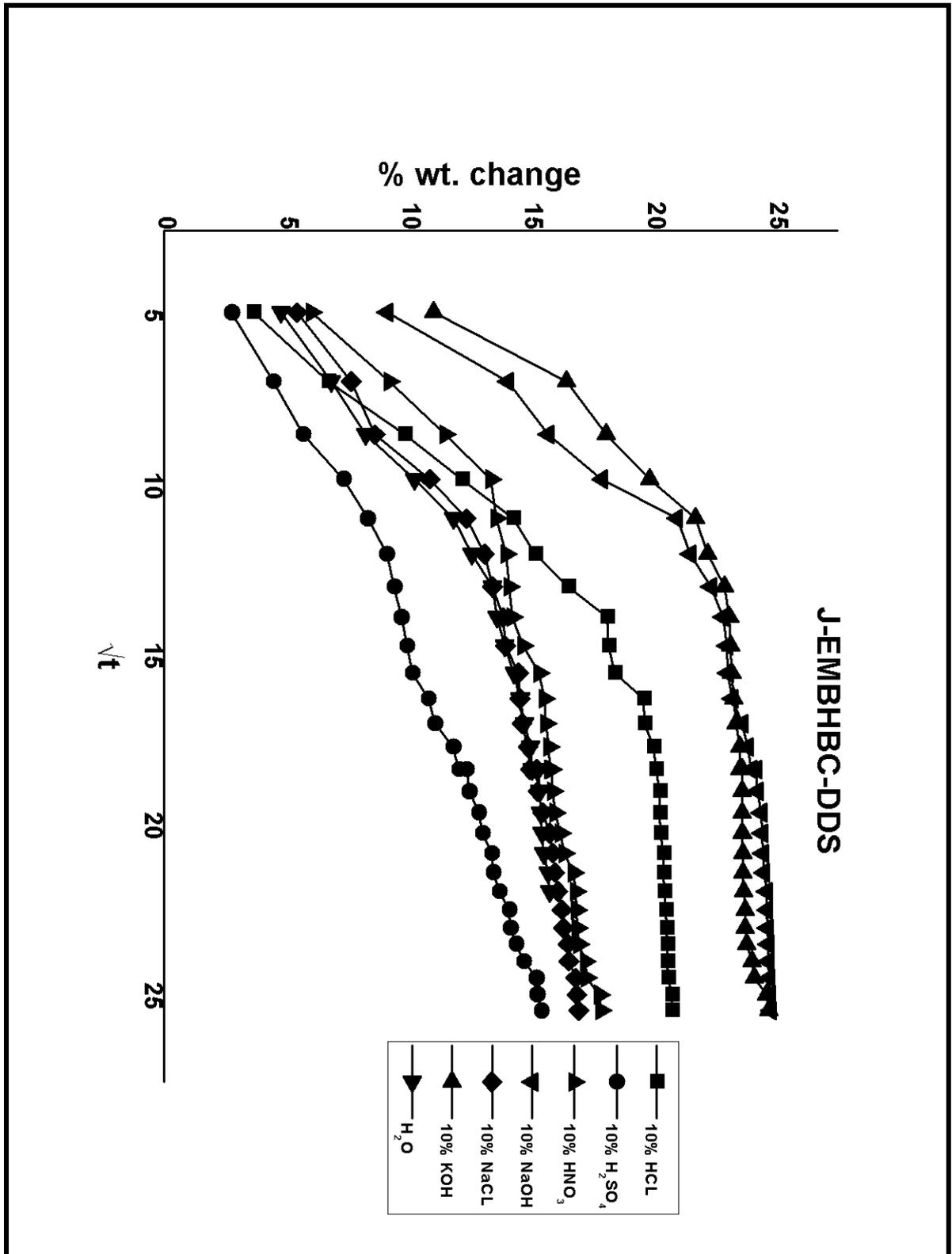


Fig. 1. The plot of % water absorbed against  $\sqrt{t}$  in different environments at 35°C for J-EMBHC-DDS.

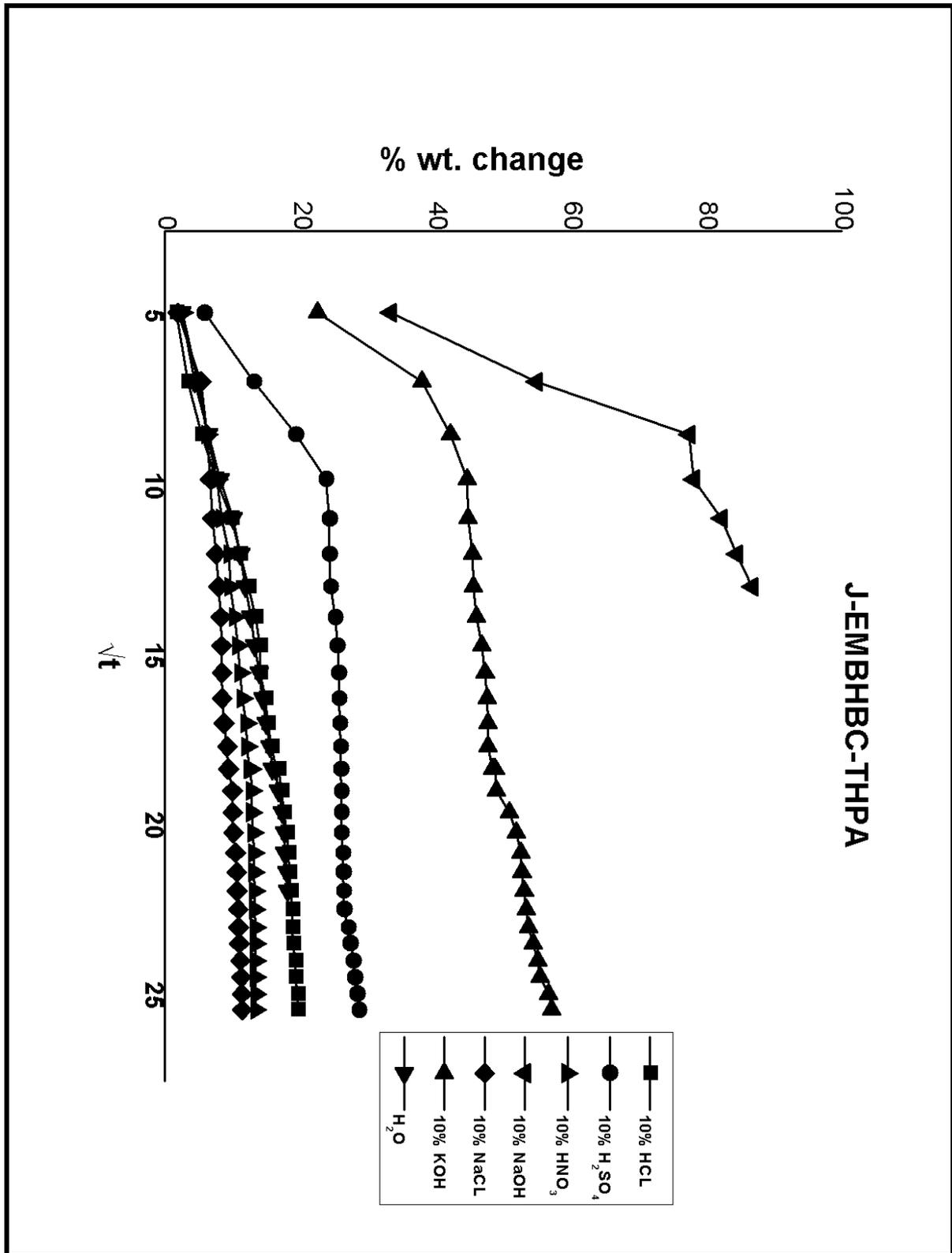


Fig. 2. The plot of % water absorbed against  $\sqrt{t}$  in different environments at 35 °C for J-EMBHBC-THPA.

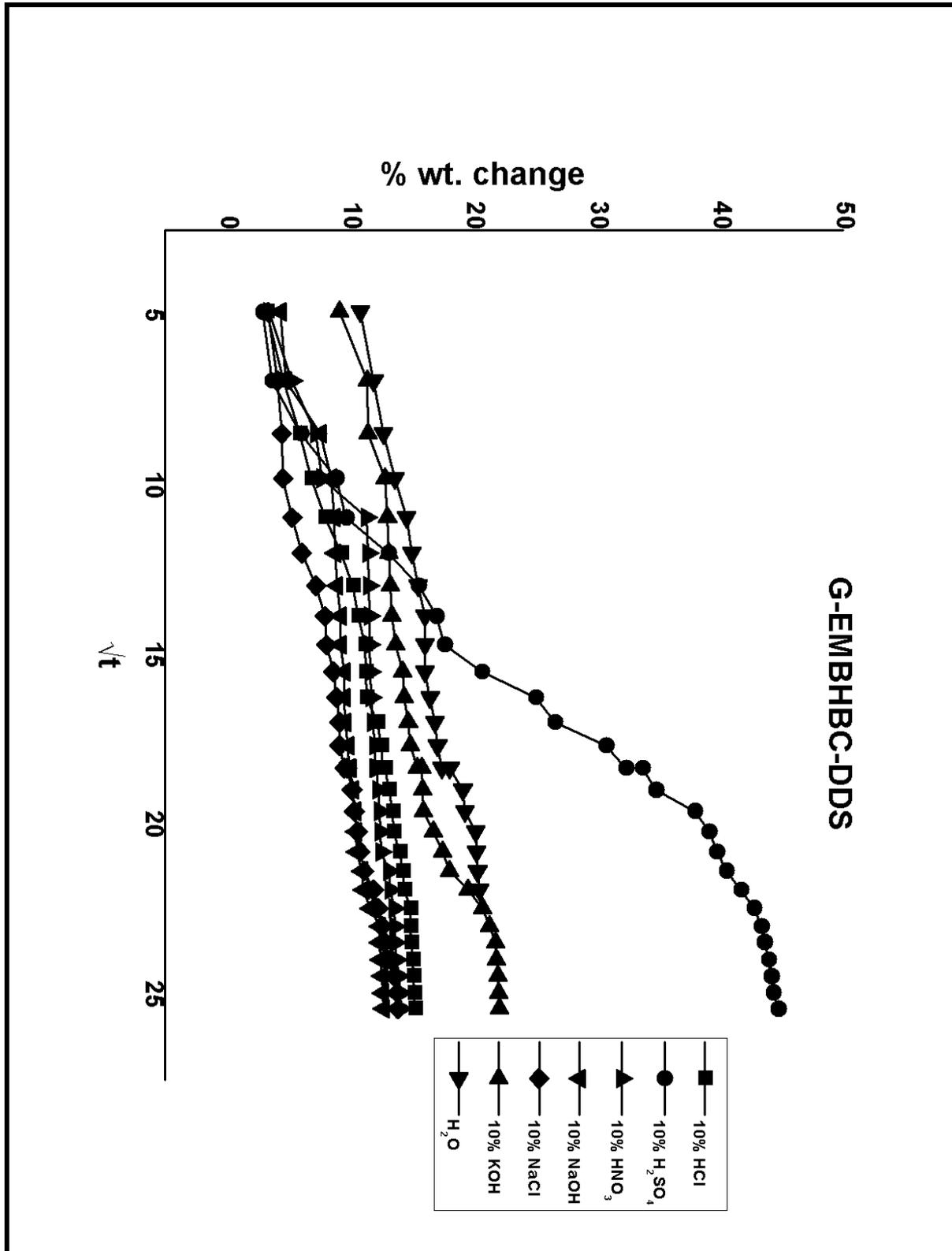
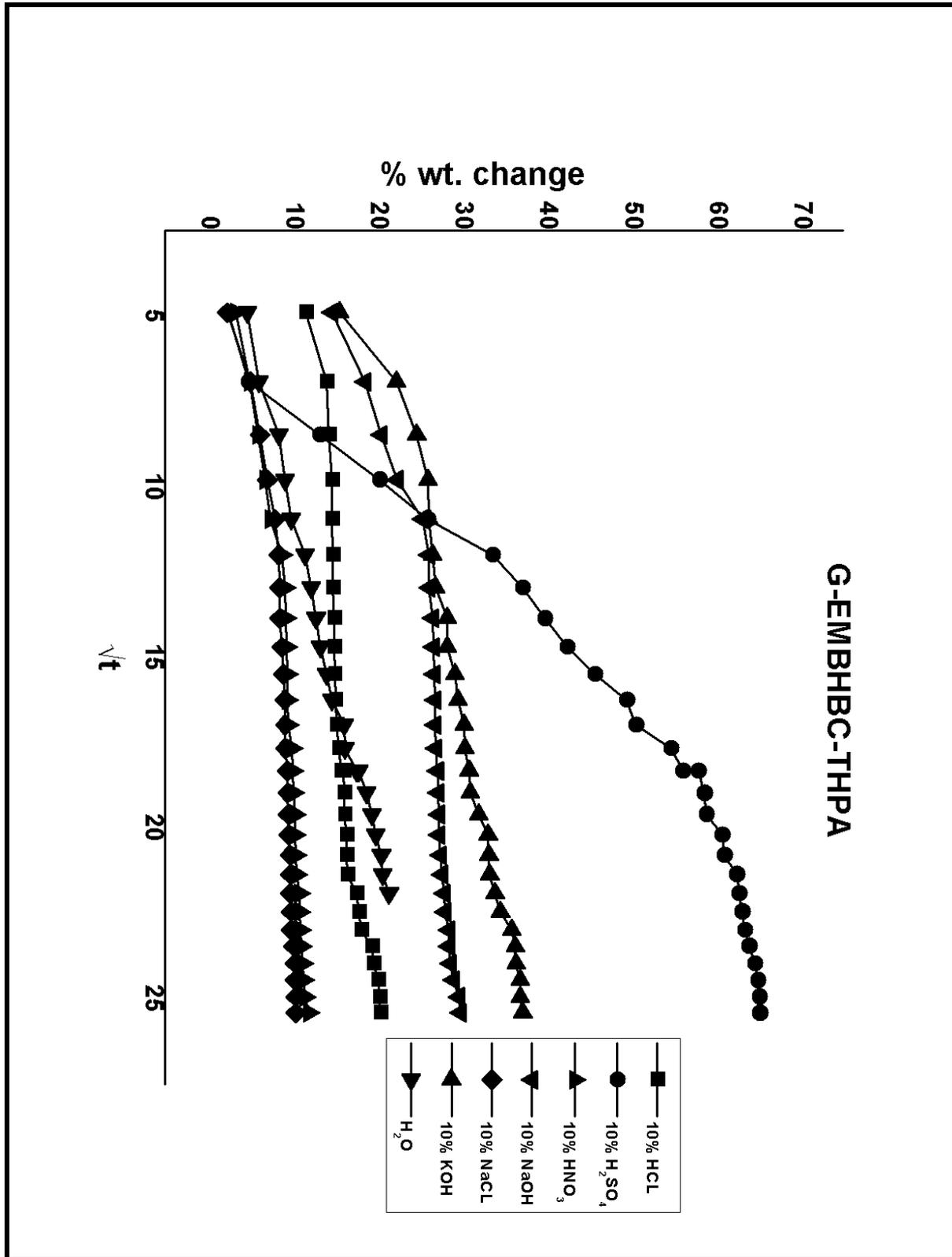


Fig. 3. The plot of % water absorbed against  $\sqrt{t}$  in different environments at 35 °C for G-EMBHBC-DDS.



**Fig. 4.** The plot of % water absorbed against  $\sqrt{t}$  in different environments at 35 °C for G-EMBHBC-THPA

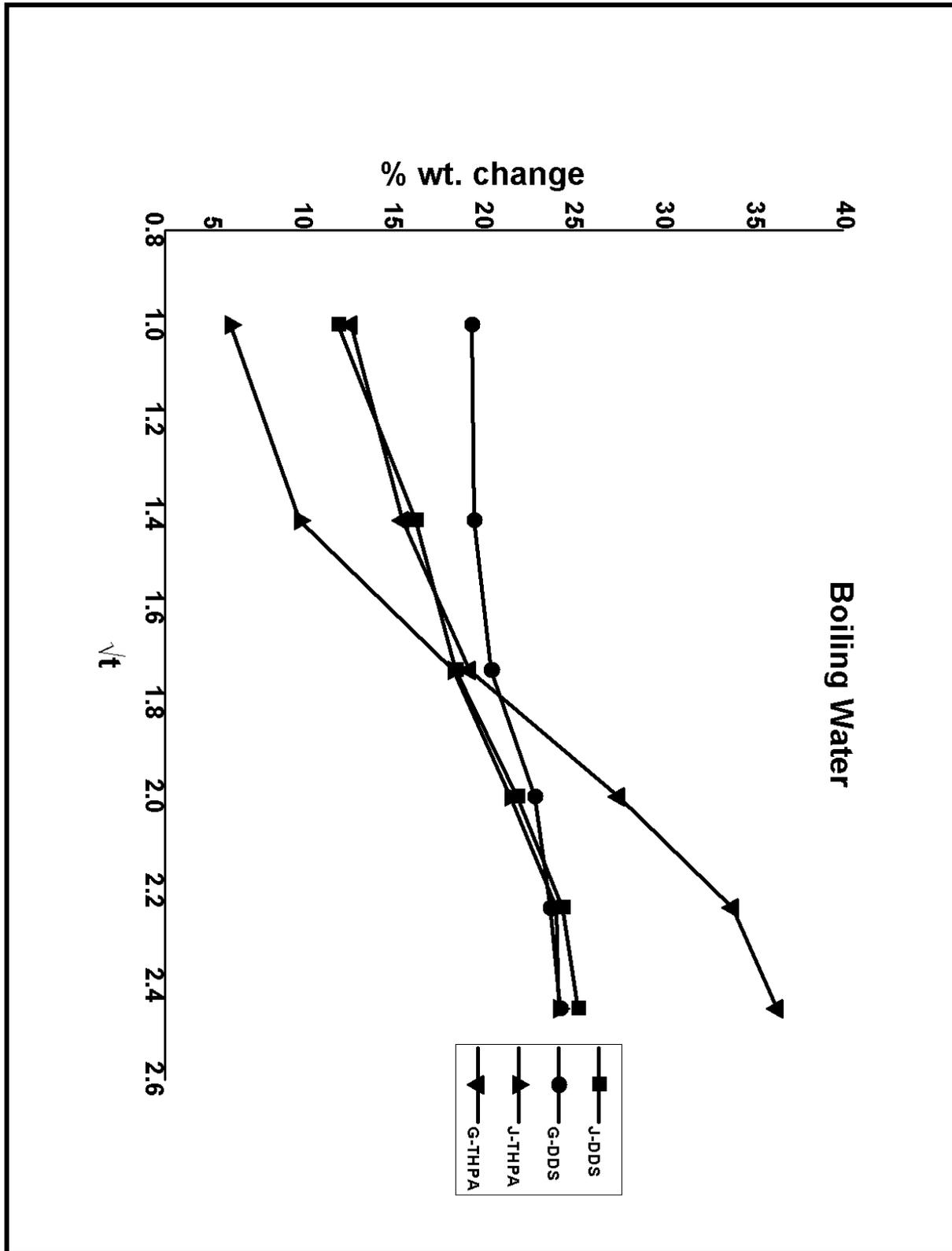


Fig. 5. The plot of %water absorbed against  $\sqrt{t}$  in boiling water for J-EMBHBC-DDS, JEMBHBC-THPA, G-EMBHBC-DDS and G-EMBHBC-THPA.