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Research Article

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Preparation and performance of low temperature epoxy resin chemical grouting agent

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ABSTRACT

Epoxy resin chemical grouting agents are widely used in the treatment of weak foundations and concrete cracks, especially the ones in alpine areas, it is important for the epoxy resin grouting agents with excellent performance at low temperature. The main agent of epoxy resin was modified by micromolecule formulating method and then a sulfhydryl modified low temperature curing agent was prepared in this paper. The epoxy resin chemical grouting agents with good groutability and curing effect were prepared and suitable for low temperature. Then the properties of grouting agents with different compositions were studied at - 8℃. This kind of grouting agent would had a widely application in treatment with weak foundations and concrete cracks at the temperature below 0 ℃.

Keywords: -8℃, Epoxy resin, Grouting agent, Preparation.

INTRODUCTION

In the north of 40°N region in China, the temperature in winter as low as -40℃, the stable period of freezing weather as long as 120d-150d, and the ice thickness reach to 1.0m . For example, Tibet region with a unique periglacial dry cold climate, which annual mean temperature is -4℃, extreme minimum temperature is -45.2℃, and 180days below 0 ℃ each year. Another similar region is Xinjiang in China. The problems such as weak foundation and concrete crack during the processes of construction or operation of water conservancy projects in alpine areas such as Tibet and Xinjiang, which require the performance of grouting agent epoxy resin suitable for the low temperature [1, 2].

As a thermosetting resin, epoxy resin was widely used as traditional epoxy grouting agents, but it was not suitable for low temperature. Because of the slow curing reaction at low temperature, long initial setting time, and difficult to solidify the concrete. Furthermore, the groutability decreased with the higher viscosity as the lower temperature. In order to suit to different working requirements of chemical grouting for different water conservancy projects, a high performance grouting agent is required at low temperature. Generally, epoxy solidification depends on activity of epoxy group and activity of active group of the curing agent, high performance grouting agent can be acquired by controlling molecular structure of the main agent of epoxy resin, preparation and formulation optimization of low temperature curing agent. In view of this, this paper focused on preparation and performance study of low temperature grouting agent, which provided significant technical support for widely application of high performance epoxy grouting agent at low temperature [3-6].

EXPERIMENTAL SECTION

Raw materials

Raw materials for the experiment including epoxy resin, curing agent, furfural, and acetone and so on are shown in table 1.

Table 1 Raw materials

Name of raw material	Type or specification	Manufactuer
Epoxy resin	CYD-128	Epoxy resin plant of Yueyang petrochemical
Acetone	Firsts	Beijing Yanshan petrochemical co.,Ltd
Furfural	Firsts	Chemical plant of zhumadian city
Curing agent	–	In house

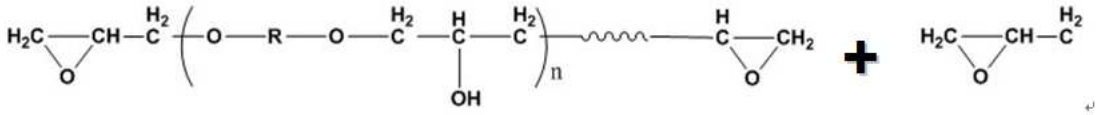
Test contents

Preparation procedure of the low temperature curing grouting agent includes selection of epoxy resin, compound modification of the main agent of epoxy resin, preparation of the low temperature curing agent and performance study of the low temperature curing grouting agent.

RESULTS AND DISCUSSION

Compound modification of the main agent of epoxy resin

The main agent of epoxy resin was further modified by micromolecule compound method, the principle is shown in Fig.1. Epoxy micromolecule was blended with the main agent of epoxy resin, a low temperature glue soluting of epoxy resin with doublet or multimodal distribution of its molecular weight was obtained [7]. Among compound glue soluting of epoxy resin, presence of epoxy micromolecule can improve infiltration capacity, thus can obtain epoxy resin with low viscosity (Table 2).



Molecule of epoxy resin **Micromolecule**

Fig.1 Schematic diagram of modification of epoxy resin by compound method

Table 2 Main performances of epoxy resin at different temperatures

Type of resin	Epoxide number	Softening point	Viscosity(mPa·s)	Organic halide value	Volatile matter
Epoxy resin for room temperature	0.41~0.47	21~20	–	＜0.02	＜1.0
Epoxy resin for low temperature	0.50~0.54	–	11000~14000	＜0.0056	＜0.5

Preparation of curing agent for low temperature

The key to achieve curing at low temperature is to select a suitable curing agent for low temperature, aliphatic amine used as curing agent currently, but it with low activity at low temperature. A novel low temperature curing agent was prepared by modifying a polymeric ketoamine curing agent with sulfhydryl, which can further improve its low temperature reaction activity [8].

The polymeric ketoamine curing agent was modified with sulfhydryl, so a sulfhydryl modified polymeric ketoamine was prepared. It was taken as the low temperature curing agent. Its IR spectrum is shown in Fig 2 and Fig 3, the peak at 2550 cm^{-1}～2590cm^{-1} is stretching vibration peak of sulfhydryl.

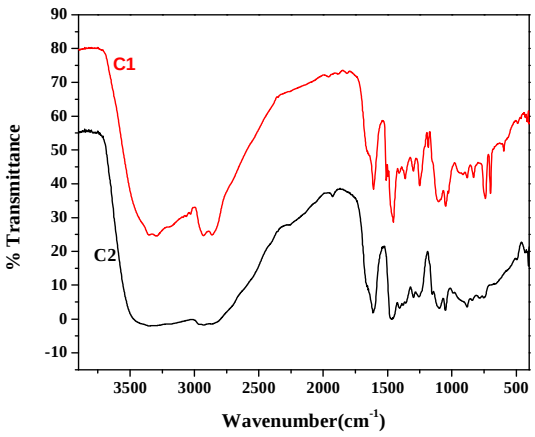


Fig.2 IR spectrum of curing agent C1 and C2

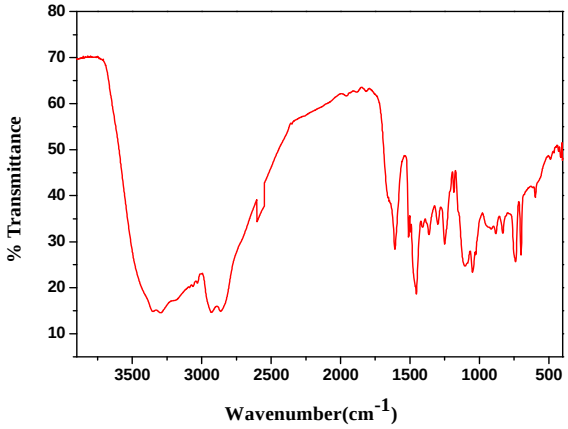
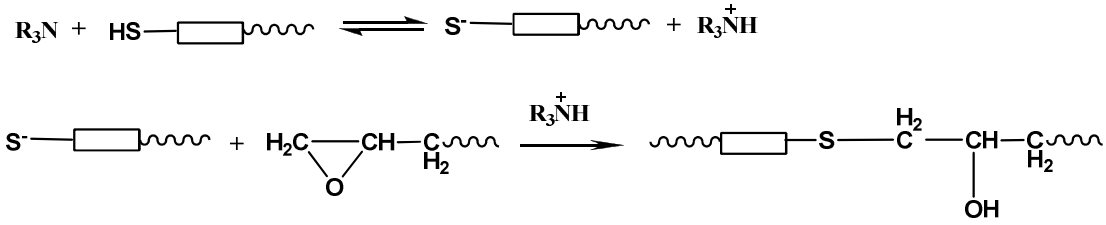
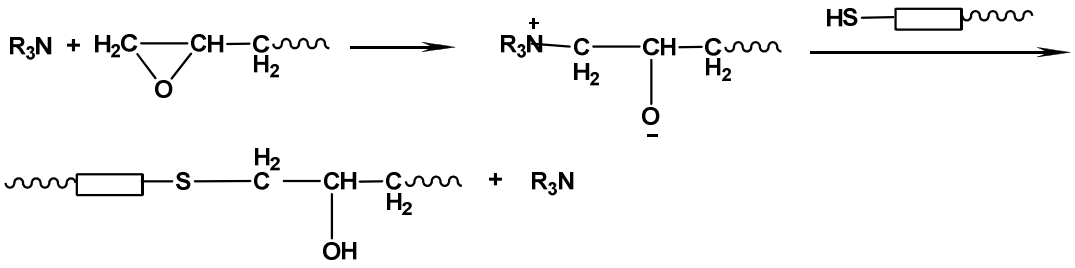


Fig. 3 IR spectrum of sulfhydryl modified polyketoamine

Meanwhile, the home-made polymeric primary amine was modified with amino group, and the acquired polymeric tertiary amine was used matching with poly-mercaptan. While the tertiary amine exists, sulfhydryl of polymeric mercaptan reacts with tertiary amine yields mercaptan ion firstly, the ion continue to react with epoxy group of epoxy resin, the reaction mechanism is expressed as follows,



Moreover, when tertiary amine reacts with epoxy group yields epoxy anion, nucleophilic reaction of the anion with sulfhydryl happens.



Selection of diluent

As the high viscosity of epoxy resin, which not suitable for directly used for grouting, so a diluent should be used to reduce its viscosity. There are three types of diluents at present, the first kind is inactive diluent, such as benzene, toluene, dimethylbenzene and acetone etc, they may be volatilized during the curing processes, thus may cause large volume contraction, and this diluent doesn't take part in the reaction of epoxy resin, its usage amount is limited, and it can limitedly reduce the viscosity. The second type is a micromolecule compound with one or more than one epoxy group, such as epoxypropane butyl ether(501#), three hydroxyl propane glycidol ether etc, they can take part in curing reaction, as these compounds have high viscosity itself, the diluent effect is poor, and restrict the groutability. The third type is diluent system of furfural-acetone, as furfural and acetone are low viscosity solvents, so this type can be taken as an effect diluent of epoxy resin before reaction, meanwhile, furfural and acetone can react with each other yield furan resin, furan resin reacts with epoxy resin yields a cross-linked structure. Its main functions are reducing the viscosity of serous fluid, improving groutability for fine crack and improving toughness of cured substances. In order to insure the groutability of grouting agent at low temperature, diluent system of furfural-acetone was adopted.

Performance of low temperature curing grouting agent

Added different amounts of surface treating agent and other additives into the low temperature epoxy resin containing a certain amount of diluent, took this as A component, took the modified low temperature curing agent as B component,

mixed component A with B and mixed well, solidified for a certain period at the temperature below 0 ℃, a epoxy resin chemical grouting agent for low temperature was acquired.

This study chose -8℃ to simulate environment of microthermal climate, at this temperature, change the ratio of component A and B, respectively tested the performances such as viscosity, operable time, compressive strength, strength of extension and bonding strength etc. From the performance test results of different curing agent ratios in table 2, it can be shown that at -8℃, curing agent have low viscosity and suitable operable time. At -8℃,when ratio of A:B was 3:1,, initial viscosity was 60mPa.s, after blended for 1h, viscosity was 89mPa·s, compressive strength of 90days age was 40.5MPa, strength of extension was 6.54MPa, bonding strength was 3.10Mpa, comprehensive performance was best. This can indicate that the prepared low temperature epoxy grouting agent have both good groutability and solidification effect at low temperature.

Table 2 Performance test results of different grouting agent components

Material ratio (A:B)		3:1	4:1	5:1
Viscosity (mPa·s)	0	60	50	60
	1h	89	64	70
	2h	＞1000	800	640
	3h	–	860	760
	4h	–	＞1000	＞1000
Gelation time (h)		87	127	320
Compressive strength (MPa)	28d	12.5	6.79	17.05
	90d	40.5	25.6	43.8
Strength of extension (MPa)	28d	3.25	2.13	1.86
	90d	6.54	3.20	2.83
Bonding strength (MPa)	28d	1.60	0.9	1.79
	90d	3.10	2.36	2.52

CONCLUSION

(1) The main agent of epoxy resin was modified by micromolecule formulating method, in the compound glue soluting of epoxy resin, presence of epoxy micromolecule can improve infiltration capacity, thus can obtain epoxy resin with low viscosity.
(2) The polymeric ketoamine curing agent was modified with sulfhydryl, so a novel low temperature curing agent was prepared, the reaction activity was further improved at low temperature.
(3) Main performances of grouting agents with different compositions were studied at - 8℃, a chemical grouting agent epoxy resin with good groutability and curing effect was prepared successfully and it is suitable for low temperature, which provided technical basis for its widely application at the temperature below 0℃.
(4) The prepared low temperature chemical grouting agent epoxy resin was applied to Eighty-one Daban Tunnel Engineering of lli river abstraction works in Xinjiang region at Mar, 2010, it successfully solved problems of concrete crack, exhibited good impermeable effect.

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