

硅树脂的绝缘性及耐热性

王 迅, 黄艳华, 孙文艳, 冯圣玉, 陈剑华

(山东大学化学与化工学院, 济南 250100)

摘要: 以一甲基硅氧烷低聚物和二甲基硅氧烷低聚物为原料, 加入适量的室温固化性能调节剂, 在催化剂作用下合成了硅树脂; 研究了催化剂种类对硅树脂性能的影响, 并研究了耐热性、固化性能与绝缘性能的关系。结果表明: 以自制催化剂制得的硅树脂的绝缘性能优于以盐酸为催化剂制得的硅树脂的绝缘性能, 且不需中和、洗涤, 简化了生产工艺; 选用两种聚甲基硅氧烷低聚物制得的硅树脂, 既可常温固化, 也可加温固化, 其耐热性较由甲基烷氧基硅烷单体聚合的硅树脂高; 固化完全的硅树脂漆膜在 200 °C 下老化 30 min 后, 绝缘电阻可保持在 1000 MΩ, 同时还具有良好的附着性和硬度, 且无毒。

关键词: 硅树脂, 硅氧烷低聚物, 绝缘性, 耐热性, 贮存期

中图分类号: TQ324.2⁺1

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有机硅材料是具有安全性、高可靠性、多功能性、多形态、高功能的绿色环保新材料。其中硅树脂可用作环保涂料、灌封材料、脱膜剂、不锈钢耐热涂层、防锈涂层、粘接剂、密封剂、绝缘材料、耐热材料等。随着应用领域的拓展, 市场对硅树脂不断提出新的要求。各种电子电气材料通常要求具有优良的绝缘性及耐热性能。作为粘接剂、涂膜材料、密封材料使用时应满足下列要求: 耐热、绝缘; 应用工艺简单, 使用方便; 单组分, 不需进行再调配; 能室温固化; 绿色环保, 在施工或使用过程中无毒、无有毒气体逸出; 贮存稳定性好, 贮存期大于 1 年^[1-3]。

笔者研究硅树脂多年, 发现以甲基氯硅烷为原料制得的无溶剂硅树脂, 其绝缘、耐高温、防水、环保性能都非常优良; 但固化温度太高, 需在 300 °C 下加热 1 h 才能固化, 使用不方便^[4]。以甲基三乙氧基硅烷为原料制得的甲基硅树脂贮存稳定性不够好。本实验探索了以一甲基硅氧烷低聚物和二甲基硅氧烷低聚物为原料合成硅树脂的方法, 以改进硅树脂的贮存稳定性及固化性能, 并重点研究了其耐热性和绝缘性。

1 实验

1.1 主要原料

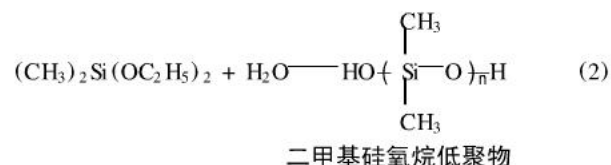
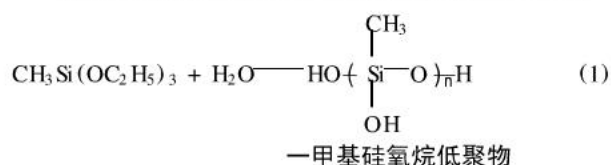
甲基三乙氧基硅烷、二甲基二乙氧基硅烷: 工业级, 浙江新安化工集团有限公司; 硅树脂

1: 由甲基烷氧基硅烷单体聚合而成, 山东国华化工新材料公司; 异丙醇: AR, 天津化学试剂二厂; 乙醇: AR, 莱阳精细化工厂; HCl: AR, 莱阳化学试剂厂; NaHCO₃: AR, 天津市泰兴试剂厂; 室温固化性能调节剂: 多官能有机硅化合物, 自制; 催化剂 ZSYS: 自制。

1.2 硅树脂的制备

1.2.1 硅氧烷低聚物的制备

硅氧烷低聚物按式 1、式 2 的合成路线制备。



一甲基硅氧烷低聚物的制备: 以甲基三乙氧基硅烷为原料, 加入控制量的去离子水和催化剂, 80 °C 下搅拌 8 h; 减压, 蒸出占原料总质量的 10%~30% 的溶剂, 备用。

二甲基硅氧烷低聚物的制备: 以二甲基二乙

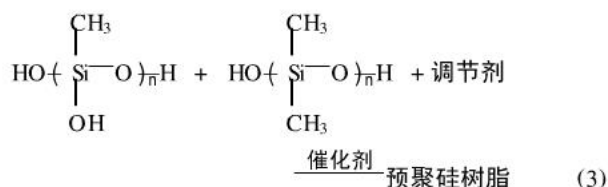
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作者简介: 王迅 (1968—), 女, 在职申请硕士, 主要研究方向为有机硅高分子。电话: (0531) 88565561。

氧基硅烷为原料,加入控制量的去离子水及催化剂,制法同上。

1.2.2 硅树脂的制备

硅树脂按式3的合成路线制备。



以ZSYS为催化剂制备硅树脂2:在带有温度计、强力搅拌器、回流冷凝管的三口烧瓶中,依次加入一定量的一甲基硅氧烷低聚物、二甲基硅氧烷低聚物、调节剂、乙醇,升温至60℃;将一定量的水和催化剂ZSYS加入到反应体系中,再升温至80℃,恒温搅拌8h;减压蒸出占原料总质量的10%~30%的溶剂,加入一定量的异丙醇,配成硅树脂溶液2。

以盐酸为催化剂制备硅树脂3:在带温度计、强力搅拌器、回流冷凝管的三口烧瓶中,依次加入一定量的一甲基硅氧烷低聚物、二甲基硅氧烷低聚物、调节剂、乙醇、去离子水和浓度为0.2 mol/L盐酸,70℃下搅拌8h;用NaHCO₃中和、洗涤,浓缩至固体质量分数为70%~90%;停止搅拌及加热,加入一定量的异丙醇,配成硅树脂溶液3。

1.3 结构表征与性能测试

红外光谱(IR):将样品烘干,采用美国Thermo Nicolet公司的Avatar 370型红外光谱仪测试;热失重分析:采用美国Perkin-Elmer公司的7 Series的热重分析仪测试,氮气氛下,升温速率20℃/min;绝缘电阻:采用杭州电表厂的ZC25-4型绝缘电阻表测试;热老化性能:在不锈钢板上涂一层硅树脂,室温放置24h,然后放入上海树立仪器仪表有限公司的101F-1型电热鼓风干燥箱中,分别在50℃、100℃、150℃、200℃、250℃下放置30min,冷却后取出,测其绝缘电阻;常温固化性能:在不锈钢板上涂一层硅树脂,室温放置,每隔5h测一次绝缘电阻,直至绝缘电阻基本稳定;热固化性能:在不锈钢板上涂一层硅树脂,放入电热鼓风干燥箱中,在分别在50℃、100℃、150℃下放置60min,冷却后取出,测其绝缘电阻。

2 结果与讨论

2.1 硅树脂的红外表征

硅树脂2的红外光谱见图1。

图1中,3432 cm⁻¹附近的宽峰为Si-OH的特征吸收峰,2975 cm⁻¹处为C-H键的伸缩振动吸收峰,1037~1117 cm⁻¹附近的宽峰为Si-O-Si中Si-O键的伸缩振动吸收峰,1274 cm⁻¹处的尖锐峰是Si-CH₃的面外弯曲振动吸收峰,766 cm⁻¹处是Si-CH₃的振动吸收峰。从图1可以看出,由二种硅氧烷低聚物合成的硅树脂中还存在着一定量的Si-OH,因此是没有反应完全的预聚物。在使用过程中其Si-OH可进一步缩合成固体硅树脂。

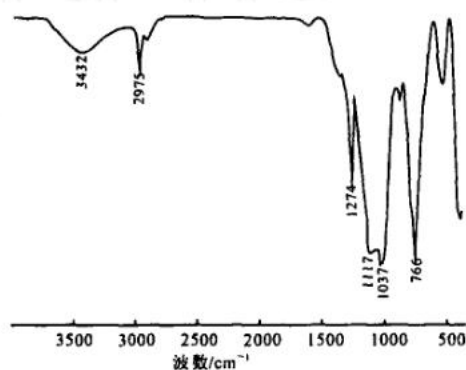


图1 硅树脂2的IR谱图

2.2 硅树脂的耐热性

图2是硅树脂固化后的热失重曲线。

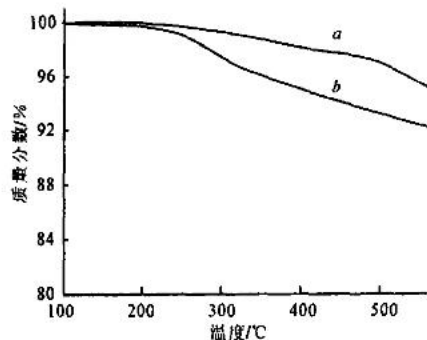


图2 硅树脂的热失重曲线

a—硅树脂2; b—硅树脂1

由图2可见,由二种硅氧烷低聚物合成的硅树脂在100~300℃之间的质量损失率只有0.552%,在300℃才开始略有失重,在300~500℃之间的质量损失率只有2.299%;而由甲基烷氧基硅烷单体聚合的硅树脂在250℃就开始失重。由此可见,由二种硅氧烷低聚物合成的硅树脂具有更高的耐热性;但数据是在升温速度为

20 °C/min的条件下得到的, 因此仅代表硅树脂的短期耐高温性。

2.3 催化剂种类对硅树脂绝缘性能的影响。

硅树脂 3 的固化膜光亮; 但绝缘性较差, 绝缘电阻在 0 MΩ 附近。而硅树脂 2 的固化膜不仅光亮; 且绝缘性优良, 绝缘电阻在 1 000 MΩ 左右。这是因为以 HCl 为催化剂时, 虽经 NaHCO₃ 中和、洗涤; 但 Cl⁻ 很难洗涤干净 (因为硅树脂中还存在羟基和烷氧基, 对水有亲和力, 过度洗涤会影响硅树脂的产率), 残存的少量 Cl⁻ 会造成硅树脂膜击穿。而用 ZSYS 做催化剂时, 不需中和, 也没有离子残存, 因此硅树脂的绝缘性比较高; 且不需中和及洗涤, 生产过程也变得简单。因此以下实验均采用以 ZSYS 为催化剂的硅树脂 2。

2.4 热老化温度对硅树脂绝缘电阻的影响

图 3 是硅树脂 2 在不同温度下老化 30 min 后的绝缘电阻与热老化温度的关系。

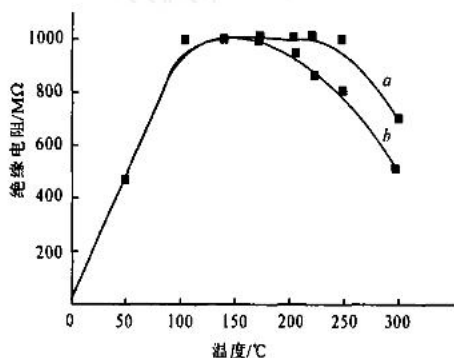


图 3 硅树脂的热老化温度与其绝缘电阻的关系
a—硅树脂 2; b—硅树脂 1

由图 3 可见, 采用二种硅氧烷低聚物合成的硅树脂经 150 °C 以上高温老化后的绝缘电阻高于采用甲基烷氧基硅烷合成的硅树脂。这可能与其结构的规整性有关。因为采用甲基烷氧基硅烷聚合时, 一甲基硅氧烷与二甲基硅氧烷的排列是无序的; 而采用二种低聚物再缩合时, 形成了嵌段共聚物, 这种结构相对于前者比较规整。事实上, 采用二种硅氧烷低聚物合成的硅树脂可在 200 °C 使用; 而采用甲基烷氧基硅烷合成的硅树脂只能在 150 °C 使用。热老化温度较低时, 因漆膜没有固化好, 所以绝缘电阻比较低; 热老化温度超过 100 °C 以后, 漆膜的绝缘电阻达到 1 000 MΩ; 当温度超过 250 °C 时, 漆膜的绝缘电阻又下降。这可能是由于温度过高时引起硅树

脂部分降解所致。

图 4 是硅树脂 2 在 200 °C 下老化一定时间后的绝缘电阻与老化时间的关系。

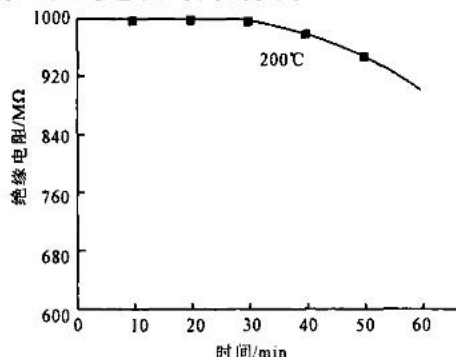


图 4 硅树脂 2 的热老化时间与其绝缘电阻的关系

由图 4 可见, 漆膜在 200 °C 下的热老化时间超过 30 min 后, 其绝缘电阻下降较快。

2.5 硅树脂的固化性能

硅树脂 2 在室温 (20 °C) 下的固化时间对其绝缘电阻的影响如图 5 所示。

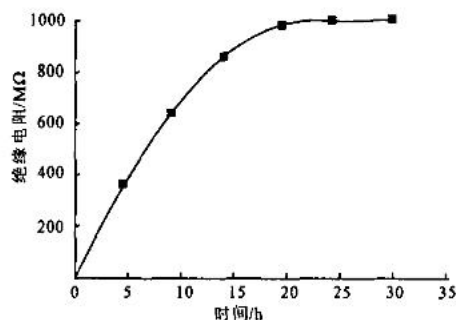


图 5 硅树脂 2 在室温下的固化时间与其绝缘电阻的关系

由图 5 可见, 硅树脂 2 在室温下固化 20 h 以后, 其绝缘电阻可稳定在 1 000 MΩ。

硅树脂 2 也可以加热固化, 其固化温度与绝缘电阻的关系见图 6。

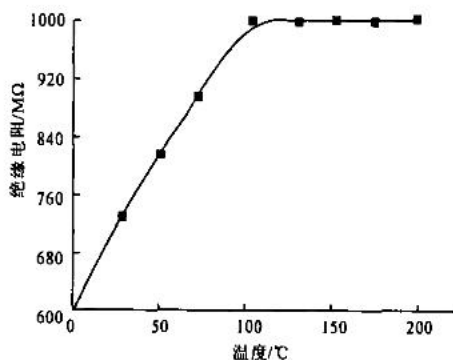


图 6 硅树脂 2 的固化温度与其绝缘电阻的关系

由图 6 可见, 当固化温度达到 100 °C 时, 漆膜的绝缘电阻基本稳定在 1 000 M Ω 。即硅树脂 2 既可室温下固化, 也可在 100 °C 固化。

2.6 硅树脂的其它性能

经山东省产品质量监督研究所测试, 硅树脂 2 的试板在 100 °C 或 200 °C 下烘 2 h 后, 漆膜的附着力 (划圈法) 均达到 1 级, 铅笔硬度均达到 6 H; 即单项判定均为合格。

经国家药品监督管理局、济南医疗器械质量监督检验中心检测, 受试动物的静脉注射样品浸提液未见急性反应, 即样品无急性毒性作用。

3 结论

以一甲基硅氧烷低聚物和二甲基硅氧烷低聚物为原料, 加入适量的室温固化性能调节剂, 在催化剂作用下合成了硅树脂; 研究了催化剂种类对硅树脂性能的影响, 并研究了耐热性、固化性能与绝缘性能的关系。结果表明: 以自制催化剂

制得的硅树脂的绝缘性能优于以盐酸为催化剂制得的硅树脂的绝缘性能, 且不需中和、洗涤, 简化了生产工艺; 选用两种聚甲基硅氧烷低聚物制得的硅树脂, 既可常温固化也可加温固化, 其耐热性较由甲基烷氧基硅烷单体聚合的硅树脂高; 固化完全的硅树脂漆膜在 200 °C 下老化 30 min 后, 绝缘电阻可保持在 1000 M Ω , 同时还具有良好的附着性和硬度, 且无毒。

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研发动态

GE 高新材料集团推出新型纺织添加剂

GE 高新材料集团有机硅部近期推出 Magnasoft ESC 和 Magnasoft NeoNYS 两款纺织柔软整理剂, 新产品可减少对环境的不良影响, 在提高织物的柔软度、蓬松感和产品性能的同时, 成本比传统的改性有机硅柔软剂降低很多。基于 GE 获专利的线性嵌段共聚物技术, Magnasoft ESC 能使织物产生极佳的丝质触感; 与同类产品相比, 用量更少; 且经多次家庭洗熨后仍可保持柔软效力。Magnasoft NeoNYS 则可赋予织物独有的干爽且柔软的手感, 可应用于各类织物而不会或极少使织物变黄。这两种产品因为不含 APEO 成分, 所以均达到目前最新的环保要求。

Magnasoft ESC 能有效覆盖织物的纤维表层并均匀渗透到内部, 所以在用量比其它有机硅柔软剂少的情况下仍可赋予织物松软、柔滑及亲水的手感; 具有水可分散性, 可应用在需要极佳剪切稳定性的后整工艺中, 包括浸渍及浸轧整理, 而不会使织物出现硅斑等质量问题。Magnasoft ESC 可用于各类纤维和织物上, 而应用在纯棉或其混纺、纤维胶人造纤维、毛织品和亚麻织品

上的效果更明显。

Magnasoft NeoNYS 是一种环氧改性的有机硅柔软剂。这种新产品的性能及手感与氨基改性有机硅柔软剂或传统的依靠机械乳化的环氧改性有机硅柔软剂完全不同。此外, 当 Magnasoft NeoNYS 与碳氟化合物同浴使用时, 能有效提高织物的柔软度, 而不会明显降低织物的防水性能。产品具有极佳的乳液及剪切稳定性, 并可改善织物的拉伸性和折皱回复性。经 Magnasoft NeoNYS 整理的 100% 全棉梭织物, 其拉伸性恢复了 93.5%, 伸长率则只有 3.05%, 而折皱恢复角为 107° (经纱) 及 83.33° (纬纱)。Magnasoft NeoNYS 可用于各类织物, 特别适用于厚重的机织物, 如牛仔布和灯芯绒等; 对人造棉纤维及聚酯纤维、仿皮织物、棉混纺及棉/弹性纤维混纺针织物等均有出色的柔软效果。

苯乙烯型环硅氧烷交联聚丙烯

华南理工大学的刘刚等人将含氢环硅氧烷与 1-辛烯在铂催化进行硅氢加成反应, 然后再与二乙烯基苯进行硅氢加成反应, 制成含乙烯基苯基的环硅氧烷; 将其作为交联剂与过氧化异丙苯一起加入到聚丙烯 (PP) 中, 能使 PP 具有一定程度的交联。

ABSTRACT

Current thinking of the development of silicone industry in China. LIU Wei-ping (Shandong Hongri A' kang Chemical Corporation, Linyi 276021, Shandong). *Youjigui Cailiao*, 2007, 21 (1): 9-13.

Abstract: The current status of silicone industry in China, major methylchlorosilane manufacturers and their capacities, as well as the key developments of several major foreign silicone manufacturers in China were reviewed. Several key points on the of silicone manufacturing capacity and overall development for the industry were suggested.

Keywords: silicone, methylchlorosilane, Xin'an, Xinghuo, Dow Corning, Wacker, GE, Shinetsu

Synthesis and characterization of octaammonium chloride salt of octaaminopropyl polyhedral oligomeric silsesquioxane.

SHEN Yuan, LI Qi-fang (College of Materials Science and Engineering, Beijing University of Chemical Technology, Beijing 100029, China). *Youjigui Cailiao*, 2007, 21 (1): 14-17.

Abstract: Octaammonium chloride salt of octaaminopropyl polyhedral oligomeric silsesquioxane (oap-POSS) was synthesized by hydrolytic condensation of γ -aminopropyltrithoxysilane in the methanol solution catalyzed by concentrated hydrochloric acid. The salt was precipitated with tetrahydrofuran. It was characterized by FTIR, ^1H NMR, ^{29}Si NMR, ^{13}C NMR and DSC. The effects of temperature, reaction time, amount of hydrochloric acid, amount of THF on yield were discussed in details. The optimal reaction conditions were as follows: the volume of γ -aminopropyltrithoxysilane, methanol, hydrochloric acid and tetrahydrofuran was 15 mL, 360 mL, 30mL, and 250 mL respectively. The reaction temperature was 90 °C and the reaction time is 18 hours. DSC analysis showed the melt point was 412.65 °C.

Keywords: cage-like, octaammonium chloride salt of octaaminopropyl polyhedral oligomeric silsesquioxane, γ -aminopropyltrithoxysilane, methanol, hydrochloric acid, tetrahydrofuran

Reaction mechanism of polycarbosilane at high temperature under high pressure (II): structure analysis of distillation products.

CHENG Xiang-zhen, XIE Zheng-fang, SONG Yong-cai, XIAO Jia-yu (State Key Lab of Advanced Ceramic Fibers & Composites, National University of Defense Technology, Changsha 410073, Hunan). *Youjigui Cailiao*, 2007, 21 (1): 18-22.

Abstract: The coarse polycarbosilane (PCS) was synthesized at 450 °C under the high pressure with liquid polysilane (LPS). The reaction mechanism of PCS was conferred from the characteristics of typical PCS structures with different molecular weight obtained by atmospheric distillation at different temperatures. The results showed that during the synthesis, Si-Si bonds of LPS were converted to Si-C bonds as the reaction temperature increased, and the low molecular car-

bosilane was first formed. As the reaction temperature further increased, the small carbosilane molecules were combined through dehydrogenation and dehydrocarbonation condensation reaction; PCS was thus obtained.

Keywords: polycarbosilane, high pressure, polycarbosilane, silicon carbide

Effects of inorganic-organic hybrid nano $\text{CaCO}_3/\text{SiO}_2$ composite particles on silicone rubber properties.

WANG Jing, LIU Guo-jun, ZHANG Gui-xia, LIU Jun-long (School of Chemistry Engineering & Material, Liaoning 116034, Dalian). *Youjigui Cailiao*, 2007, 21 (1): 23-28.

Abstract: $\text{CaCO}_3/\text{SiO}_2$ composite nano particles with core-shell structure were prepared by sol-gel precipitation process, and were organically in-situ hybridized. The $\text{CaCO}_3/\text{SiO}_2$ composite nano particles was used as amplifying material to replace partial silica particles in silicone rubber. The mechanical properties and the thermal stability of silicone rubber thus prepared were characterized by SEM, pulling experiments machine and heat loses machine. The $\text{CaCO}_3/\text{SiO}_2$ nano particles modified with different in-situ organic reagents further improved the mechanical properties and thermal stability of silicone rubber. Compound particle hybridized with A-151 showed the most improvement, while compound particle hybridized with KH-570 showed less improvement. It was also found that the mechanical properties and thermal stability of silicone rubber were also affect by the amount of $\text{CaCO}_3/\text{SiO}_2$ composite nano particles. When less than 10 % of the silica particles were substituted with KH-570 hybridized compound particle, the performances of silicone rubber were better than the silicone rubber modified by the fumed silica.

Keywords: organic hybrid, nano CaCO_3 , silicone rubber, fumed silica, silane coupling agent

The electrical resistivity and heat resistance of silicone resin.

WANG Xun, HUANG Yan-hua, SUN Wen-yan, FENG Sheng-yu, CHEN Jia-hua (Shandong University, Jinan 250100, Shandong). *Youjigui Cailiao*, 2007, 21 (1): 29-32.

Abstract: The silicone resin was prepared by condensation polymerization of methyltriethoxysilane oligomer and dimethyldiethoxysilane oligomer in the presence of ZSYS as catalyst. The effect of silicone properties using various catalysts and the relation among the thermal stability, electrical resistivity, cure at the normal temperature and the storage time were studied. Results indicated that the silicon resin prepared by these two silicone oligomers could be cured both at room temperature and higher temperatures; their thermal resistance is better than the resin prepared by dimethylchlorosilane monomers. The weight loss of these resins began at 300 °C. The weight loss increased at 300 ~ 500 °C, and the total weight loss was 2.299 %. However, the weight loss of the resin prepared by dimethylchlorosilane

monomers began at 250 °C. the electrical resistivity of the resin film, which fully cured and aged at 200 °C for 30min, could reach 1000MΩ. The non-toxic film also showed good adhesion and hardness.

Keywords: silicone resin, siloxane oligomerization, electrical resistivity, thermal stability, storage time

Synthesis of amino silicone fluid with high viscosity. SU Yu-guang (Quzhou Chemical Group, Quzhou 324004, Zhejiang). *Youjigui Cailiao*, 2007, 21 (1): 33-35.

Abstract: A new synthesis method of low molecular weight α , ω -dihydroxy polydimethyl siloxane and *N*- β -aminoethyl- γ -diethylaminopropylmethyldimethoxy silane and method of viscosity control were introduced. The results indicated that the viscosity of the amino silicone fluid prepared by this method was higher than traditional method, and the reaction temperature was lower than traditional method. The fabric treated with silicone micro emulsion had excellent hand feel and smoothness.

Keywords: amino silicone fluid, α , ω -dihydroxy polydimethyl siloxane, *N*- β -aminoethyl- γ -diethylaminopropylmethyldimethoxy silane

Preparation and application of amino and dodecyl co-modified polysiloxanes. HUANG Liang-xian, AN Qiu-feng, LI Ming-tao, YANG Gang, WANG Qian-jin, FU Yong-shan (College of Chemistry and Chemical Engineering, Shanxi University of Science & Technology, Xiangyang 712081, Shanxi). *Youjigui Cailiao*, 2007, 21 (1): 36-40.

Abstract: In the presence of tetramethylammonium hydroxide (TMAH) as catalyst, a new softener amino and dodecyl co-modified polysiloxanes (ADMPS) was synthesized by bulk polymerization of octamethylcyclotetrasiloxane (D4), *N*- β -aminoethyl- γ -aminopropylmethyldimethoxysilane (YDH-602), dodecylmethyldimethoxysilane (HD-109) and hexamethyldisiloxane (MM). The chemical structure of ADMPS was characterized by IR and ¹H NMR. It was applied for the conditioning of 100 % white cotton. The results showed that when the amino value of ADMPS was increased, the bending rigidity of the treated cotton decreased and its wrinkle recovery angle increased, which enhanced soft hand feel, the elasticity and the antirease performance of the treated cotton. As the alkyl value of ADMPS was increased, the softness, elasticity and the antirease performance of the treated cotton declined a little, and the whiteness and wettability unchanged. The results showed that at similar amino content, the wettability and smoothness of cotton treated with ADMPS (alkane value 0.3mmol/g) was better than that with *N*- β -aminoethyl- γ -aminopropylpolydimethylsiloxanes (ASO-1), the whiteness of treated cotton fabric was as the same as that treated by ASO-1, whereas the softness and wrinkle recovery angle of the cotton treated with ASO-1 was better than that treated with ADMPS.

Keywords: *N*- β - α - γ -diethylaminopropylmethyldimethoxy silane, polysiloxanes, amino silicone fluid, dodecyl polysiloxanes, textile, softening agent

Analysis of methylchlorosilanes & phenylchlorosilanes by GC & GC-MS with micro-column. JIANG Ke-zhi, NI Yong, JIANG Jian-xiong, QIU Hua-yu, WU Ji-rong, LAI Guo-qiao (Key Lab of Organosilicon Chemistry and Material Technology of Ministry of Education, Hangzhou Teachers College, Hangzhou 310012, Zhejiang). *Youjigui Cailiao*, 2007, 21 (1): 41-43.

Abstract: The complete separation of chlorosilane mixture of phenyltrichlorosilane, dimethyldichlorosilane, methyltrichlorosilane and methyl phenyldichlorosilane were realized with BN-200ms micro-column, which was confirmed by GC-MS.

Keywords: phenyltrichlorosilane, dimethyldichlorosilane, methyltrichlorosilane, methyl phenyldichlorosilane, micro-column, GC-MS, GC

The latest application development of polysiloxane modified polyurethane. CHEN Xue-juan, ZHU Jie, HUANG Shi-qiang (Faculty of Material Science and Engineering, Hubei University, Wuhan 430062, Hubei). *Youjigui Cailiao*, 2007, 21 (1): 44-47.

Abstract: The latest research and application development of polysiloxane modified polyurethane in the field of polymer dielectric, biomedicine and membranes materials etc were reviewed.

Keywords: polysiloxane, polyurethane

Research progress in synthesis of 1,1,1,3,5,5,5-heptamethyltrisiloxane. WU Lu-yan, ZHAN Xiao-li, CHEN Feng-qiu (UNILAB Research Center of Chemical Reaction Engineering, Zhejiang University, Hangzhou 310027, Zhejiang). *Youjigui Cailiao*, 2007, 21 (1): 48-52.

Abstract: The synthesis methods of 1,1,1,3,5,5,5-heptamethyltrisiloxane (MD^HM) were reviewed. Then the reaction mechanism of hydrolysis and condensation polymerization of methylchlorosilane was analyzed. In the end, future direction of preparation of MD^HM in the future was pointed out.

Keywords: heptamethyltrisiloxane, methylchlorosilanes, hydrolyzation, condensation

The blending technology of mixed silicone Rubber (VII). HUANG Wei-run (Chenguang Research Institute of Chemistry Industry, China Bluestar, Chengdu 610041, Sichuan). *Youjigui Cailiao*, 2007, 21 (1): 53-57.

Abstract: The blending technology of four types of silicone rubber compound for automobile, i. e. axial seal and connecting shaft protective casing, gaskets for engine system and waterproof connector, were introduced

Keywords: silicone rubber, automobile, axial seal, gasket, oil resistivity, fatigue resistance, self-lubricity