

C IR 高效减水剂的合成

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摘 要 C IR 古马隆树脂系高效减水剂, 成本低, 性能好, 具有广泛的应用前景。本文介绍了其合成原理、合成方法、反应控制因素及成品对混凝土性能的影响。

关键词 古马隆树脂减水剂 合成原理 合成方法 混凝土性能

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我国自 70 年代起开始研制、生产与应用高效减水剂, 至今已有二十多年的历史, 取得了显著的成就。如今高效减水剂的用途已日益广泛, 用量也逐年增长, 但由于种种原因, 目前国内市场使用的高效减水剂仍主要是萘系产品^[1]。根据权威机构的预测, 世界焦炭总产量在未来 10 年呈平稳趋势^[2], 作为炼焦副产品的工业萘产量自然也不会增长, 所以随着高效减水剂需求量日益增大, 工业萘原料会越来越紧缺, 价格也会不断上涨。故开发非萘系高效减水剂, 扩大高效减水剂的生产原料来源, 有着重要意义。

1 C IR 的合成

1.1 古马隆树脂

古马隆树脂俗名煤焦油树脂, 是以 160 ~ 190 煤焦油馏分(除古马隆和茚组份外, 还含有一定量的甲基氧茚、二甲基氧茚、双环戊二烯、苯乙烯等)为原料在催化剂存在下经溶液聚合而得, 产品为暗褐色无定型固体, 分子量一般在 2 000 左右^[3], 我们研究和应用的古马隆树脂来自武钢焦化有限公司。

1.2 合成原理简述

C IR 的合成主要经历磺化反应和中和反应, 同时伴有聚合反应和水解反应, 产品性能好坏主要取决于磺化反应和中和反应。

1.2.1 磺化反应 古马隆树脂的磺化是一个亲电取代的可逆平衡反应, 反应情况极为复杂。首先, 古马隆树脂的苯环上有 α 位和 β 位之分, α 位电子云密度较大, 比较容易磺化, 磺化物也比较容易水解, 而 β 位电子云密度较小, 较难磺化, 磺化物也较难水解^[4]。由于磺化结束后, 物料粘度较大, 必须加大量的热水稀释后才能再加碱和石灰中和, 所以希望磺化反应能尽量在 β 位上而不在 α 位上进行, 使树脂磺化物不因补水稀释时发生水解反应, 降低物料磺化度, 影响产品性能, 而要想尽可能多的生成 β 位磺化物, 就需较多的硫酸用量、较高的反应温度和反应时间^[4]。但硫酸既可作古马隆树脂的磺化剂, 也是古马隆树脂继续聚合的催化剂, 因此在磺化阶段维持较高的反应温度和反应时间来减少 α 位磺化物生成的同时, 会加大古马隆树脂间的聚合反应, 会生成大量分子量高于 5 000、水溶性差、分散性不好的聚合产物, 而且聚合反应会使反应物粘度急剧增加, 甚至固化, 容易造成生产设备事故。故古马隆树脂磺化时, 必须选择加入一种合适的反应助剂(一种酚类阻聚剂), 达到既可抑制古马隆聚合反应, 又可尽量多的生成 β 位磺化产物, 提高产品性能的目的。

1.2.2 中和反应 古马隆树脂磺化结束后, 物料粘度很大, 需先用热水稀释到一定含量才有利于加碱中和。加热水稀释时, 磺化反应产生的少量 α 位磺化树脂会发生水解反应, 重新生成古马隆树脂和硫酸。

由于磺化时加入了过量的硫酸, 水解时也生成了一部分硫酸, 因此中和若全部采用液体烧碱, 则最终产品会含有超过 35 % 的硫酸钠, 大大降低产品性能, 使产品难以在高强、高性能混凝土中应用。故中

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和须采用液碱和石灰乳两步中和, 通过用石灰乳中的氢氧化钙与硫酸生成硫酸钙沉淀, 将产品中硫酸钠含量降低到 5% 以下。但要想有效、经济的去除硫酸钙沉淀, 则需选用高速离心的除渣设备, 而不宜选用传统的板框压滤机。

1.3 CR 的合成方法

1.3.1 将大块古马隆树脂稍作破碎处理后, 计量投入反应釜中, 加入反应助剂, 升温熔融后, 开搅拌, 于一定温度下, 细流加入浓硫酸, 恒温进行磺化反应。

1.3.2 磺化结束后, 稍降温, 慢加热水稀释, 加完热水后, 搅拌 15 min 左右, 加液碱和石灰乳中和至物料 pH 值为 7~10, 注意加水和加碱速度不可太快, 防止冲料。

1.3.3 将中和好的物料进行过滤, 除去硫酸钙沉淀, 水剂物料可根据需要, 浓缩至一定含量, 作水剂成品使用, 也可干燥成粉剂产品, 粉剂成品呈棕褐色。

1.4 合成主要控制因素对 CR 分散性能的影响

CR 合成的主要控制因素在于硫酸加入量、反应助剂加入量、磺化温度与磺化时间。为了找到比较好的合成控制因素值, 利用正交试验表 L₉(3⁴) 进行多因素优选法试验。见表 1、表 2。

表 1 正交试验选用的合成控制因素与水平

因素 水平	A 硫酸与树脂质量比	B 反应助剂与树脂质量百分比/%	C 磺化温度/ /	D 磺化时间/h
I	1.2 1	3	130	1
II	1.5 1	5	150	1.5
III	2.0 1	8	170	2.0

表 2 正交试验结果

编号	A	B	C	D	物料状态	流动度 ²⁾ / /mm
1	I	I	I	II	极稠	90
2	I	II	II	III	较稠	160
3	I	III	III	I	较稠	150
4	II	I	II	III	较稀	205
5	II	II	III	I	较稀	230
6	II	III	I	II	较稀	220
7	III	I	III	I	较稀	208
8	III	II	I	II	较稀	225
9	III	III	II	III	较稀	240
I ₁ ¹⁾	400	503	535	588		
I ₂ ¹⁾	655	615	605	535		
I ₃ ¹⁾	673	610	588	605		
I ₁ '(I ₁ /3)	133 3	167.7	178 3	196		
I ₂ '(I ₂ /3)	218 3	205	201.7	178 3		
I ₃ '(I ₃ /3)	224 3	203 3	196	201.7		
R _i ¹⁾	91	37.3	23.4	23.4		

注: 1) I₁、I₂、I₃ 分别为 I、II、III 水平的试验指标(流动度值)总和, R_i 为最好水平与最差水平之差值;

2) 流动度采用 GB 8077—87 方法, 用中国华新 525# 硅酸盐水泥, 每次水泥用量为 300 kg, 自来水 87 g, CR 有效掺量为 1.8 g。

从试验结果和试验时的物料状态分析: 硫酸加入量不够时, 不利于 β 型树脂磺酸盐的生成, 造成物料磺化度不够, 粘度变大, 产品分散性低, 流动度值也很低; 反应助剂的加入, 主要是抑制古马隆树脂在磺化时继续聚合生成高分子量物质, 降低产品性能。反应助剂品种选择正确与否及加入量是否合适, 在某种程度上决定 CR 的合成能否顺利进行, 及能否得到较好的产品性能; 磺化温度较低时, 可能会生成大量的 α 型树脂磺化物, 这些物质在加水稀释时, 又会重新水解成树脂和硫酸, 严重影响产品分散性, 而温度太高可能不利于反应助剂充分发挥作用, 也易造成部分物质脱水磺化; 反应时间以树脂能充分磺化为宜, 不可太短。根据正交试验结果, 得出最好水平组合为 A_{III}B_{II}C_{II}D_{III}, 即硫酸加入量为树脂重量的 2 倍, 反应助剂为树脂重量的 5%, 磺化温度为 150, 磺化时间为 2 h。

2 CR 的减水机理

CR 的主要成份为磺化古马隆树脂钠盐。磺酸盐类物质一般易溶解于水, 溶解后均有离解作用, 生成带负电荷的有机阴离子(R⁻)及金属阳离子, 因此它属于阴离子表面活性剂, 离解后的阴离子两端性质不同, 一端为能溶于油而难溶于水的亲油基团, 为有机烷链; 另一端是难溶于油而易溶于水的亲水基团(磺酸基)。随着烷链的加长, 它在水中的溶解性将变差^[4,5]。

水泥加水转变成水泥浆后, 由于: 粒子间的范德华引力作用; 水泥初期开始形成架状水泥水化矿物; 水泥主要矿物在水化过程中带不同电荷而互相吸引, 使水泥浆在微观上是一种絮凝状结构, 这种结构中包裹了不少水^[1]。当 CR 掺入水泥浆中, CR 的有机阴离子借助于分子间的引力和水泥颗粒作用, 被定向吸附到水泥颗粒表面, 亲油端与水泥颗粒相连, 亲水端朝向水溶液, 形成单分子层或多分子层的吸附膜, 这种效果是拉拢了水分子而隔开了絮凝状的水泥粒子, 从而释放出絮凝体中被包裹的水分子。同时由于有机阴离子的定向吸附, 使水泥颗粒朝外一侧带有同种的负电荷, 产生了相斥作用, 因而使水泥浆形成了一种稳定的分散体系。因而水泥浆中加 CR 后, 能够产生增加分散性, 减少拌合用水量的效果^[1,5]。

3 掺 CR 的混凝土性能

3.1 试验材料

湖北华新 525 普硅水泥, 中国一冶 525 普硅水泥, 湖北三峡 525 普硅水泥, I 级粉煤灰(湖北阳逻电厂), 超细矿粉(武钢生产, 比表面积 5 500 cm²/g), 硅灰(武钢生产), 粗集料 5 mm ~ 25 mm (湖北武穴田镇

石厂), 细集料 m_x = 2.9 (河砂)。

3.2 C IR 与萘系减水剂 FDN 性能对比试验

试验按照 GB 8076—1997 进行, 水泥选用中国一冶 525 普硅, 试验结果见表 3。

表 3 C IR 与 FDN 性能对比实验

编号	减水剂 C × %	单方砼用量/kg				W / C	坍落度 /mm	减水率 /%	平均强度与抗压强度比/%		
		C	S	G	W				R3	R7	R28
1	0	330	765	1 150	198	0.60	71	0	16.9/100	22.8/100	31.4/100
2	C IR 0.4	330	765	1 150	167	0.506	83	15.7	25.2/149	29.9/131	38.6/123
3	C IR 0.6	330	765	1 150	156	0.473	90	21.2	27.0/160	33.7/148	47.1/150
4	C IR 0.8	330	765	1 150	146	0.442	90	26.3	30.6/181	38.1/167	47.1/150
5	C IR 1.0	330	765	1 150	142	0.430	79	28.3	31.6/187	39.9/175	48.0/153
6	FDN 0.4	330	765	1 150	168	0.509	90	15.2	24.5/145	31.7/139	41.1/131
7	FDN 0.6	330	765	1 150	157	0.476	85	20.7	28.6/169	35.6/156	46.2/147
8	FDN 0.8	330	765	1 150	147	0.445	75	25.8	36.3/215	43.1/189	48.7/155
9	FDN 1.0	330	765	1 150	139	0.421	88	29.8	37.8/224	44.4/195	53.0/169

注: 表中 C、S、G、W 分别表示单方混凝土的水泥、砂、石、水的用量, R3、R7、R28 表示混凝土 3 天、7 天、28 天的养护龄期, W / C 表示水与水泥的用量之比。

从表 3 的试验数据看出, C IR 的掺量增加, 混凝土的水灰比减小, 减水率和抗压强度值增加, 与本公司 FDN 对比数据说明, C IR 的产品性能接近于目前使用最广泛的萘系高效减水剂。

3.3 C IR 配制高强混凝土

试验使用的碎石经过清洗处理, 配合比中使用部分细掺合料取代水泥, 胶结料(包括水泥和掺合料)的总用量较大, 见表 4。

表 4 C IR 配制高强混凝土试验结果

编号	强度等级	每立方砼材料用量/kg					C IR 掺量(对胶结料)/%	坍落度 /mm	平均强度 / MPa	
		C	S	G	掺合料	W			R3	R28
1	C60	一冶水泥 470	660	1 080	粉煤灰 20	170	1.0	230	51.30	71.8
2	C70	一冶水泥 480	625	1 120	矿粉 75	155	1.1	225	56.5	84.1
3	C80	华新水泥 465	605	1 130	硅灰 35 矿粉 65	149	2.35	1.3	61.6	93.9

试验结果表明, C IR 对两种水泥的适应性均好, 配制的混凝土抗压强度值也满足设计要求。

3.4 C IR 配制高工作性混凝土

试验采用 C IR 与缓凝剂复配, 双掺磨细矿粉和

一级粉煤灰, 配制的混凝土粘聚性好, 不泌水、离析, 混凝土具有高的流动性, 坍落度经时损失也很小, 可以满足有高工作性要求的混凝土施工。试验结果见表 5。

表 5 C IR 配制高工作性混凝土试验结果

编号	每立方米砼材料用量/kg						外加剂及掺量 (对胶结料) /%	坍落度/mm			平均强度 / MPa	
	C	S	G	W	粉煤灰	矿粉		初始	60 min	150 min	R7	R28
1	三峡 525 440	700	1 045	160	55	55	C IR 1.2 柠檬酸 0.08	235	235	220	58.4	77.8
2	三峡 525 440	700	1 045	157	55	55	C IR 1.2 磷酸盐 0.12	230	235	230	56.1	77.2
3	华新 525 440	700	1 045	162	55	55	C IR 1.2 柠檬酸 0.08	235	230	225	59.5	78.1
4	华新 525 440	700	1 045	158	55	55	C IR 1.2 磷酸盐 0.12	240	240	230	57.7	76.9

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4 结 论

4.1 CIR 是一种非萘系高效减水剂, 由于在合成的磺化过程中选择了合适的反应助剂, 在中和过程采用了液碱和石灰乳两步中和法, 选用了先进的高速离心除硫酸钙沉渣设备, 突破了原有古马隆减水剂的合成工艺, 使生产过程更加容易控制, 设备使用更加安全, 产品性能更加优良, 它的投产可缓解目前存在的萘原料紧缺局面。

4.2 CIR 产品性能接近萘系高效减水剂的水平, 在 0.5% ~ 1.0% 掺量范围内, 减水率相应可达 15% ~ 28%, 减水效果明显; 同时在一定掺量范围内使用 CIR 的混凝土强度增长幅度较大, 有较好的促进混凝土强度发展的作用。

4.3 CIR 与试验选用的三种水泥及粉煤灰、矿渣、硅灰等掺合料有很好的相容性, 可配制低水灰比, 高

流动性的高强混凝土, 而且与缓凝剂同时使用, 双掺粉煤灰、矿粉, 可配制低水灰比, 高流动性, 低坍落度经时损失的高工作性混凝土。

4.4 CIR 的吨原料费用不到 2000 元, 大大低于萘系减水剂的原料成本, 而两种减水剂的性能较接近, 因此 CIR 的生产会有明显的经济效益和社会效益。

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Optimization of the Synthesis Gas Component for Acquiring the Unit Potentials

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Abstract The theory of the methanol synthesis is reviewed and the importance of the synthesis gas component is also presented. The author proposed three kinds of preferred process on the modification of the synthesis gas component through comparison of several kinds of process on synthesis gas.

Key words methanol synthesis, natural gas, coal gas, technical revamping

Progress in Methanol Chemical Industry

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Abstract Methanol is the foundation stone of C_1 chemical industry. The production and consumption of methanol at home and abroad is analyzed and forecast in this paper. Progress in raw material route and catalyst and new process of methanol synthesis is summarized respectively. Suggestions on developing big single series of production facilities and paying attention to the research work of liquid phase synthetic technologies are given.

Key words methanol, market, raw material route, catalyst, new process

Characteristics and Progress in 2000 of Ambient Temperature Fine Sulfur-removal New Technology

Kong Yuhua Wang Xianhou Wang Guoxing
(National Key Base of CO₂ WGS and Gas Purification Catalyst, Gas Purification Center of Hubei Research Institute of Chemistry 430074)

Abstract The paper introduces the characteristics and newest application progress in protection of CO₂-removal, ammonia synthesis and H₂-removal catalyst.

Key words ambient temperature fine sulfur-removal, new technology, characteristics, process

Industrial Application of QSJ-01 COS Ambient Temperature Hydrolysis Catalyst

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Wang Dongmei Fu Yuansheng
(Research Institute of Qilu Petrochemical Corp. 255400)

Abstract Use QSJ-01 ambient temperature carbonyl sulfide (COS) hydrolysis catalyst to remove COS from methanol feed gas and present the analysis results of discharged catalyst after application. Results of application show QSJ-01 catalyst has better COS converting activity,

higher strength and COS remained in the accurately desulfurized gas was less than 0.05×10^{-6} . Thus the life-span of methanol synthesis catalyst is prolonged, and obvious economic results are obtained for the factory.

Key words COS, hydrolysis catalyst, industrial application

Brief Study on Market and Production Situation of Pitch-based Carbon Fiber

Zhang Shaocheng
(Shanxi Research Institute of Chemical Industry 030021)

Abstract Through analysis of market on carbon fiber, this paper reviewed the necessity of developing the industry of pitch-based carbon fiber, and production situation was introduced as well.

Key words pitch, carbon fiber, pitch-based carbon fiber

Process Conditions Optimum for the Additive Preparation of Coal-water Slurry with Black Liquor

Li Hanxu Bai Chengzhi Wang Qunying Li Aiyuan
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Abstract A new method, cross test, is used to improve the process of utilizing black liquor to prepare additive for coal-water slurry. The effects of reaction temperature, reaction time and the quantity of NA, H and NH etc on the additive properties are discussed. Based on the experiment and linear analysis, the optimum process conditions were obtained. The properties of coal-water slurry prepared by the additive and other two additives are compared.

Key words pulping black liquor, additive, process conditions, cross test

Study on Tensile Strength of Coke

Chen Qihou Yang Junhe
(East China Institute of Metallurgy 243002)

Abstract The relationship of coke tensile strength with R_{max} , V_{daf} , G of coal quality from single coal was studied. The results show that there exists curved line among them (coke tensile strength of medium rank coal has a higher value). The relationships of coke tensile strength to structural strength, microstrength and gasifying content for single coal were also studied. The results indicate that there exists clear relativity among them.

Key words coke, tensile strength, gasifying content

Synthesis of CIR High Range Water Reducing Agent

Zhu Huaxiong
(Haoyuan Chemical Co. Ltd of Wuhan Iron and Steel Corp. 430082)

Abstract CR coumdrone-indene resins of high range water reducing agents features in low cost and good properties and has a broad perspectives in application. Its synthetic principle, process, factors on reaction control and effects of the product on concrete performance are described.

Key words coumdrone-indene resins of water reducing agent, synthetic principle, process, concrete performance

Property Research and Progress of the New VST in China

Du Peiheng Wang Rongliang
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Du Jianting
(Tianjin University 300072)

Abstract This paper summaries the fluid mechanics property and the research of mass transferred property, the advanced progress of the New VST (New Vertical Sieve-tray Tower) in our country is also expounded.

Key words New Vertical Sieve-tray Tower, fluid mechanics property, mass transfer property

Development on the Math Models of the Heterogeneous Photocatalysis Reactors

Wang Jinwen Sun Yanping
(Taiyuan University of Technology 030024)

Abstract This article introduces the main existing methods and achievements in the field of the math models of reaction kinetics and reactor of the heterogeneous photocatalysis reaction system. Models of kinetics are analyzed according to the mechanism and the kinetics equation, moreover, the reactor models are classified by the various methodologies adopted by different academic groups and according to the situations of phases inside different reactors, the reactors can be classified into several types, this work also introduces the radiation energy distribution equation and forecasts the direction of future research in this aspect.

Key words heterogeneous, photocatalysis, kinetics, math model

Plan on the Developing of New Product of Lanzhou Coal Gas Plant

Yan Jun Zhang Fang
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Abstract The paper discusses the technique and economic possibility of the methanol coproduction by the coal gas and chemical product from methanol.

Key words coal gas, coproduction, methanol

Comparison between Three Desulfuration and Decyanation Units in COG Refining Plant of Bao Steel

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Abstract There are three desulfuration and decyanation units in COG Refining Plant of Bao Steel Phase 1 is NCS's Takahax-Hirohax process Phase 2 is NKK's Sulfiban process Phase 3 is OGE's Fumaks-Ruodacs-Compacts process. By operating several years, these three units show its merit and shortcoming. The text compares and assesses these three units one by one.

Key words desulfuration & decyanation, activator, efficiency of desulfuration

Reasons and Improving Measures of the Lower First-rated Ammonium Sulphate Yield

Liang Yi
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Abstract The reasons of the lower first-rated ammonium sulphate yield in Shanxi Coking Co., Ltd are analyzed, and the foreign substance occurred in the waste liquid of ammonium sulphate is confirmed to mainly result in the lower yield, and the relevant improving measures are described.

Key words wasteliquid of ammonium sulphate, crystallization, saturation

Process on the Disposal of Phenol-cyanogen Sewage by A/O Method

Cui Baohua Liu Jun Cao Yi Li Hongjun
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Abstract Biological denitrogenation theory, A/O technological process, mire taming and operation management were discussed in this paper. It also described how to adopt the techniques to dispose the high concentration distilling-ammonia sewage. All the indices of disposed sewage had met the GB 13456-92 draining criterion, except COD exceeded a little.

Key words biological denitrogenation, A/O method, mire taming

Analysis and Measures on the Causes of Low Crystallization Point of Coking Benzene

Chen Min Guo Chunling Li Qinghui
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Abstract Through analysis on the causes of low crystallization point, the main factors which are influential to the crystallization point of coking benzene have been obtained. It shows that the contents of satisfied hydrocarbon and CS₂ are exorbitant. The crystallization point has been risen by taking corresponding separation measures, ensuring the first rate of coking benzene.

Key words crystallization point, purification, satisfied hydrocarbon, separation efficiency