

第七届混凝土外加剂应用技术专业委员会年会

聚羧酸系高性能减水剂及其 应用技术新进展—2015

New Development in Polycarboxylate Superplasticizer and
Application Technology—2015



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前 言

随着建筑技术的发展、施工技术要求的不断提高及混凝土技术的迅速发展,我国聚羧酸系高性能减水剂及其应用技术水平不断提高。聚羧酸系高性能减水剂具有掺量低、保坍性能好、收缩率低、分子结构可调性强、高性能化潜力大、生产工艺清洁化等突出优点,其发展态势及应用前景具有可持续性。

聚羧酸系高性能减水剂的优点在工程界已被广为认可,并已成为配制高性能混凝土的首选减水剂。目前,该减水剂在铁路、公路、建工、港工、海工、水电、核电、市政工程等许多重点项目中得到广泛应用。

混凝土外加剂应用技术专业委员会已分别于2005年、2007年、2011年和2013年在北京成功举办了四届全国聚羧酸系高性能减水剂及其应用技术交流大会,在全国混凝土及外加剂等相关领域引起了强烈的反响,加快了该技术和产品的大力推广应用,促进了行业技术水平的提高。

为了更好地交流国内外聚羧酸系高性能减水剂最新应用技术与研究成果,总结近两年来工程应用的经验,推动混凝土外加剂技术水平的进步,促进业内及相关领域人士之间的交流与沟通。中国建筑学会建筑材料分会混凝土外加剂应用技术专业委员会于2015年6月16—17日在北京举办“全国第五届聚羧酸系高性能减水剂及其应用技术交流会”暨“混凝土外加剂应用技术专业委员会2015年年会”。特此编辑、正式出版了名为《聚羧酸系高性能减水剂及其应用技术新进展—2015》的论文集,本届会议论文集共收录了50篇学术价值较高的论文,主要内容涉及国内外聚羧酸系高性能减水剂在混凝土工程中的应用技术,综述与理论研究,合成、复配与性能研究,新单体、新品种与新工艺的研究,检测技术等内容。本论文集可供混凝土与外加剂领域的技术、生产、施工、质检、科研人员及大专院校师生等参考。

由于时间及水平有限,论文集中难免有不妥之处,谨请读者赐教指正。

混凝土外加剂应用技术专业委员会
2015年3月

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Current Trends in PCE Superplasticizer Technology

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1 Introduction

Concrete with a 20% reduction in water content at a given strength is a significant improvement in concrete technology. The use of superplasticizers (SP) has become a key technology for achieving this goal. The development of PCE superplasticizer technology has been a major focus of research and development in the field of concrete technology.

综述与基础理论

The main objective of this paper is to review the current trends in PCE superplasticizer technology and to discuss the basic theory of PCE superplasticizer. The paper is divided into two main parts: the first part discusses the current trends in PCE superplasticizer technology, and the second part discusses the basic theory of PCE superplasticizer.

2 Microstructure of PCE polymer

The microstructure of PCE polymer is a key factor in determining its performance. The microstructure of PCE polymer is determined by the chemical structure of the polymer and the way it is synthesized. The microstructure of PCE polymer is a complex structure, and it is difficult to describe it in detail. However, it is generally accepted that the microstructure of PCE polymer is a network structure, and it is this network structure that gives PCE polymer its unique properties.

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Current Trends in PCE Superplasticizer Technology

Prof. Dr. Johann Plank *

(Technische Universität München Chair for Construction Chemistry)

1 Introduction

Invented in 1981 and first commercialized in 1986, PCE Superplasticizers have become the most successful family of concrete admixtures. For 2014, the total quantity of PEC solution (30% solid content) produced worldwide is estimated at 3.5 million tons. This quantity even exceeds those for lignosulfonate and naphthalene sulfonate formaldehyde polycondensate. Further growth is predicted for the coming years.

In view of the high importance of PCEs for modern concrete technology it is no surprise that the industry continues to improve the fundamental understanding of these products and optimizes their performance further. In the following, some major recent findings and results will be presented.

2 Microstructure of PCE polymers

It is well established that different methods in the synthesis of PCE copolymers can result in products which exhibit different microstructures and therefore interact and perform with cement in different ways. For example, in the synthesis of MPEG type PCEs statistic, random or gradient copolymers can result, depending on the feeding method of the monomers. Similarly, in the preparation of HPEG or IPEG PCEs, acrylic acid which is considerably more reactive than the macromonomer can undergo homopolymerization as a side reaction. Therefore, the resulting PCE copolymers may have significantly different molar compositions when compared with the feeding ratio. Consequently, a method is needed which allows to determine the actual sequence of monomers in a PCE molecule.

^{13}C NMR spectroscopy has been proven to provide this possibility. For example, when the carbonyl carbon present in the carboxylate groups of methacrylic acid in MPEG PCE is selected as the trigger atom, then its chemical shift varies with the neighboring monomers. The chemical shift is

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lower when linked to another methacrylic acid monomer, and it is slightly higher when connected with the macromonomer.

As an example for this microstructure analysis, the ^{13}C NMR signals of the carbonyl carbon present in acrylic acid obtained for an IPEG type PCE are displayed in Fig. 1. There it is shown that the arrangement AAE (A = acrylic acid, E = ether macromonomer) presents the most frequent monomer combination whereas the structural units AAA or EAE are considerably less frequent. As a result, one can say that this polymer includes a statistical (irregular) distribution of the macromonomer along the main chain, and that significant blocks of homopolymerized acrylic acid are contained as well. Such blocks are favorable for adsorption on cement and thus enhance its performance as superplasticizer. This example demonstrates that knowing the actual microstructure of PCE products allows to understand their performance and to target even more improved products in future synthesis.

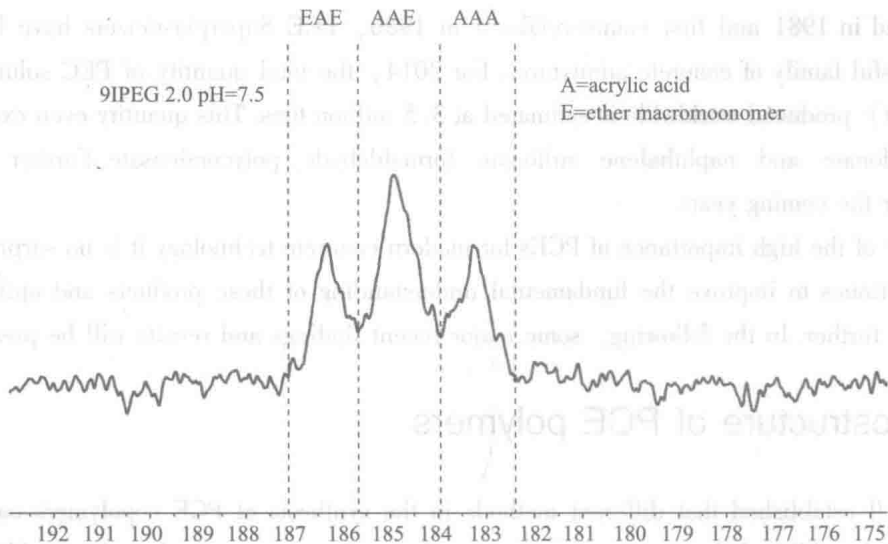


Fig. 1 ^{13}C NMR signals of the carbonyl carbon of an IPEG PCE synthesized at a molar ratio of 2 : 1 (acrylic acid; macromonomer) and a side chain length of 9 EO units

3 Cement Compatibility of PCEs

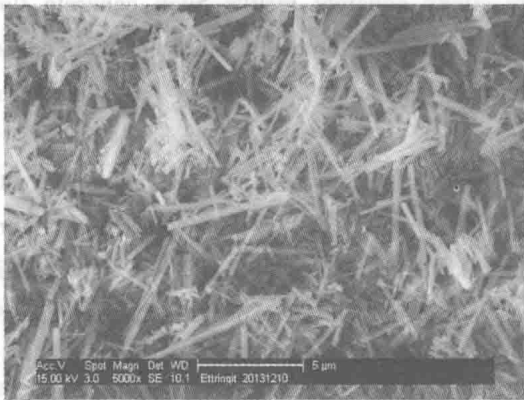
It has been reported numerously that specific cements are very difficult to disperse with PCEs. Apparently, the composition of these cements somehow prohibits the PCEs to become effective as dispersants.

In a recent study we have investigated more than 20 cements with a wide variation in their compositions, especially regarding the C_3A content which varied from 1.0% to 9.8%. For those cements, significantly different dosages of each PCE sample were required to achieve effective dispersion (see Table 1). Based on these results, two particularly “difficult” cements (CEMI

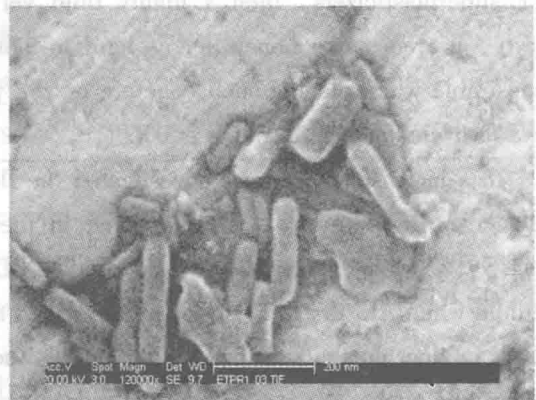
32.5R and CEM I 42.5R) requiring especially high dosages were selected and studied further. We found that incompatibility between a cement sample and a specific PCE product always occurs when (a) the cement possesses a relatively high C3A content ($\geq 6\%$) and (b) when the PCE polymer modifies the size of the early ettringite crystals to become only a few hundred nanometers long, instead of $\sim 2\mu\text{m}$ for common ettringite (Fig. 2).

Table 1 PCE dosages required for different cement samples to achieve a 26 cm slump flow for the cement paste (W/C ratio = 0.3)

Cement sample	MPEG - 7	MPEG - 25	APEG - 34	APEG - 34AM	IPEG - 25
	[PCE dosages in% by weight of cement]				
CEM I 32.5 N	0.25	0.11	0.21	0.10	0.10
CEM I 32.5 R	> 1.00	> 1.00	> 1.00	0.25	0.51
CEM I 42.5 N	0.35	0.16	0.29	0.14	0.13
CEM I 42.5 R	> 1.00	> 1.00	> 1.00	0.22	0.35
CEM I 42.5 R HS	0.15	0.09	0.14	0.08	0.08
CEM I 52.5 N	0.22	0.12	0.20	0.12	0.10
CEM I 52.5 R	> 1.00	0.90	0.45	0.31	0.26
CEM I 52.5 R HS	0.31	0.12	0.30	0.12	0.11
API Glass C	0.10	0.06	0.11	0.05	0.05



(a)



(b)

Fig. 2 Ettringite crystals precipitated in the absence of PCE (a) and in the presence of a MPEG type PCE (b); Take note of different scale bars

Hence, it became apparent that many PCE products are morphological catalysts for the growth of ettringite. This means that through adsorption on specific surfaces of the early ettringite nuclei they prevent their further growth. The result are large amounts of very short ettringite crystals which possess a large surface area which needs to be covered by PCE superplasticizers to achieve high fluidity. This effect leads to excessively high dosages of PCEs which are felt as “incompatibility” between a specific cement and a particular PCE sample. Generally, PCE polymers which do not modify the

crystal size of ettringite such as e. g. PCE samples IPEG - 50 or APEG - 34 AM as shown in Table 2, are more robust with different cements than other PCE molecules.

Table 2 Correlation between the size of early ettringite crystals produced in the presence of different PCE copolymers and their cement compatibility

PCE polymer sample	Size of ettringite crystals/nm	Compatibility with "difficult cements"
MPEG - 7	718 ± 14	poor
MPEG - 45	981 ± 24	poor
APEG - 23	1 238 ± 27	medium
IPEG - 50	1 936 ± 86	good
APEG - 34	2 050 ± 65	good

This study demonstrates that understanding the crystal growth and the surface chemistry of ettringite is of high importance. For this reason we have investigated the growth of ettringite under the undisturbed, idealized conditions of zero gravity. These results will be reported in the conference in another paper.

4 Fast-flowing PCE concrete

Applicators are well aware that when concrete of low W/C ratio (e. g. < 0.4) is admixed with PCE superplasticizers, then a highly fluid yet very sticky concrete results. Such concrete flows slowly, it creeps like honey and is hard to move (e. g. pump). Obviously, this behavior is most undesirable when placing such concrete on the job site. As a result, the industry has searched for ways to overcome this negative effect.

In a large study we have investigated the flow behavior of mortars admixed with different PCE polymers with respect to their flow time in the flow line and the empty time from the V-funnel (Fig. 3). We found that generally MPEG type PCEs produced the slowest flow. IPEG PCEs performed medium while APEG PCEs consistently provided the fastest flowing mortars. Apparently, the impact of a PCE product on the speed of mortar flow is more linked to its chemical class than to the specific composition such as e. g. molar ratio of monomers (grafting density) and length of the side chains.

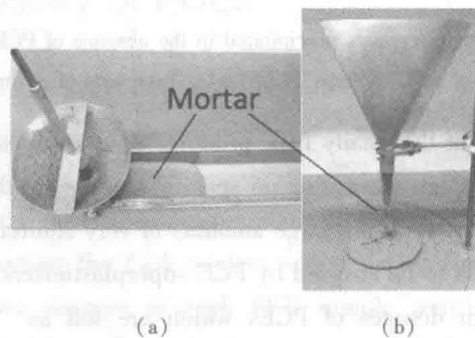


Fig. 3 Experimental setup of the flow line (a) and the V-funnel test (b)

Based on this finding we then calculated the HLB (hydrophilic-lipophilic balance) values for the PCE polymers which we had tested (Table 3). From there it became obvious that MPEG PCEs which produce slow flow speed exhibit low HLB values of between 16.5 ~ 18.0 (i. e. they are less hydrophilic than other PCEs) while the APEG PCEs which impart fast flow possess particularly high HLB values of 19.3 ~ 19.6. The results signify that PCE molecules which are most hydrophilic present the best type of superplasticizer for concretes mixed at low W/C ratio. Furthermore, as cause for the slow speed of flow a high plastic viscosity of the cement paste was identified (Table 3).

Table 3 Chemical classification, dosages, HLB values, V-funnel empty times in mortar and yield stress and plastic viscosity of cement pastes holding the PCE samples studied

PCE polymer sample	Macromonomer	PCE dosage / (% bwoc)	HLB value	V-Funnel empty time/s	Yield stress /Pa	Plastic viscosity / (mPa · s)
Blank	—	—	—	No flow	> 100	> 10 000
MPEG - 7	methacrylate ester	0.45	16.5	121	5.0	1 806
MPEG - 10	methacrylate ester	0.27	16.7	100	6.1	1 550
MPEG - 25	methacrylate ester	0.27	18.0	102	6.9	1 388
APEG - 23	allyl ether	0.35	19.3	85	6.5	1 296
APEG - 34	allyl ether	0.36	19.4	51	4.1	1 146
APEG - 34OH	allyl ether	0.36	19.5	41	5.2	1 089
APEG - 34AM	allyl ether	0.18	19.6	61	6.3	1 130
IPEG - 52	isoprenyl ether	0.18	18.8	85	7.7	1 275
IPEG - 70	isoprenyl ether	0.19	18.8	110	7.1	1 354
IPEG - 90	isoprenyl ether	0.20	18.9	100	6.9	1 298

In the meantime, results from job site applications have confirmed the findings from our mortar tests. Additionally, it was found that addition of a second polymer which does not adsorb can also increase the flow speed of concrete. This study is still ongoing and its results will be reported later.

5 State of the Art—Concrete Mixing Plant

In the following, a description of a typical ready-mix concrete plant in Germany and how they use PCE admixtures is given.

Such concrete plants produce ~200 000 m³ of concrete per year, with significantly less activity during the winter period than in summer. Nowadays, more than 80% of the concrete delivered belongs to the consistency class of F4, i. e. the spread flow on the shock table is 55 ~ 58 cm. About 10% of the concrete delivered is self-compacting concrete (SCC) with a spread flow of ~70 cm. This product requires a very careful mix design and quality control tests are conducted before it leaves the plant and can be delivered.

The concrete plant possesses two separate concrete mixers with 5 m^3 volume. The raw materials (cement, sand, aggregates) are dosed automatically from a silo into the mixer. The plant manager operates the dosing of the compounds through a remote control unit and can follow the feeding into the mixer via a camera which is installed in the mixer. Admixtures such as PCEs are fed in from 2 m^3 tanks holding the liquid admixtures. PCE is generally added $\sim 2 \sim 3$ seconds after the water has been dosed in, and mixing time for PCE concrete is 45 seconds. Recently, there has been a trend to replace common PCEs with a new generation of PCEs which adsorb very fast and thus require significantly shorter mixing times ($10 \sim 20$ seconds only). Those PCEs contain phosphate as anchoring group and therefore have higher affinity to the surface of cement than traditional polycarboxylates.

A typical German ready-mix plant consumes ~ 600 tons of liquid PCE per year which is delivered by truck in 20 m^3 tanks. Lignosulfonates, naphthalene sulfonates and melamine sulfonates are no longer used much. In the summer, $\text{K}_4\text{P}_2\text{O}_7$ is admixed as a set retarder for high temperatures while sodium gluconate is applied less. In the recent winters which were not as cold as usual, no accelerator was necessary.

Concrete trucks returning from delivery must be washed and cleaned before taking a new load. The wash water is separated into water, settled fines (cement) and aggregates. The fines and aggregates are dried and reused for low quality concrete while the wash water is filtered before it can be released into the sewage.

6 Outlook

PCE superplasticizers greatly contribute to modern concrete technology. They allow us to formulate highly durable concretes of low W/C ratio, yet with excellent workability properties. This has helped much to improve the quality of concrete which is produced nowadays.

In the future, PCE products will retain their dominant position as a key admixture for modern advanced concrete. This is because of their excellent performance and their relatively low cost, considering the complicated chemistry involved in their manufacturing chain. One can expect that in the future, both higher performing as well as even more economic PCE products will emerge on the market.

聚羧酸高性能减水剂技术的现状及发展趋势

Johann Plank

(慕尼黑工业大学无机化学系, 德国慕尼黑)

(本论文由雷蕾博士翻译)

摘要: 根据聚羧酸高性能减水剂的研究现状, 系统阐述了本课题组近期主要的研究成果, 其中包括应用 ^{13}C 核磁共振技术确定 PCE 分子的微观结构、PCE 与水泥相容性问题的机理分析, 以及从分子结构角度解释不同 PCE 混凝土流动性能的差异。最后, 笔者提出今后聚羧酸技术的发展方向, 并认为聚羧酸高性能减水剂产品仍将保持其作为现代先进混凝土的重要组成部分及核心地位。

关键词: 聚羧酸系高性能减水剂; 微观结构; 水泥相容性; 纳米级钙矾石

1 序言

自 1981 年发明、1986 年首次产业化至今, 聚羧酸高性能减水剂 (PCE) 已成为混凝土外加剂中最重要和最关键的品种。2014 年, PCE 母液 (固含量 30%) 全球总产量约 350 万吨, 远远超过木质素磺酸盐和萘系减水剂。预计在未来几年, 其产量将持续增长。

在探讨 PCE 产品对当今混凝土技术发展的重要性时, 不难发现混凝土行业一直致力于更透彻地分析 PCE 产品的作用机理并不断优化其性能。本课题组近期主要的研究成果如下。

2 PCE 聚合物的微观结构

试验证明, 不同的合成方法会生成不同微观结构的聚羧酸系高性能减水剂产品, 从而导致其相互作用和性能的表现方式的差异。例如, MPEG 系列 PCE 高性能减水剂的聚合数据显示, 无规共聚物和梯度共聚物可以通过调节聚合单体的加料方式制得。同样, 在 IPEG 和 HPEG 系列 PCE 高性能减水剂的制备过程中, 丙烯酸的聚合活性远高于大单体, 故会发生丙烯酸均聚的副反应。因此, 最终 PCE 共聚物中聚合单体的摩尔比与加料的摩尔比会有很大区别。所以, 用于表征 PCE 高性能减水剂分子中实际单体序列的方法极其重要。

经验证, ^{13}C 核磁共振 (NMR) 图谱可以实现这种可能性。例如, 将 MPEG 系列 PCE 高性能减水剂中甲基丙烯酸所含的羰基碳作为目标原子, 其化学位移将随其相邻单体的变化而变化。当相邻单体为甲基丙烯酸时, 羰基碳的化学位移会降低, 相邻单体为大单体时, 其化学位移会略高。而 IPEG 系 PCE 高性能减水剂也有类似的规律。例如, 图 1 为 9IPEG 2.0 PCE 高性能减水剂 (酸醚比为 2.0, 支链长度为 9 个 EO) 的 ^{13}C 核磁共振谱图。图中显示,

AAE (A = 丙烯酸, E = 醚类大单体) 序列含量较 AAA 或者 EAE 更高, 为含量最高的结构序列。该聚合物的分子结构为醚类大单体无规排列于丙烯酸主链上, 同时含有大量的利于超塑化剂分子吸附在水泥颗粒上的丙烯酸共聚物。确定 PCE 聚羧酸高性能减水剂的实际微观结构有助于理解其性能表现, 并通过调整合成工艺来提高产品的性能。

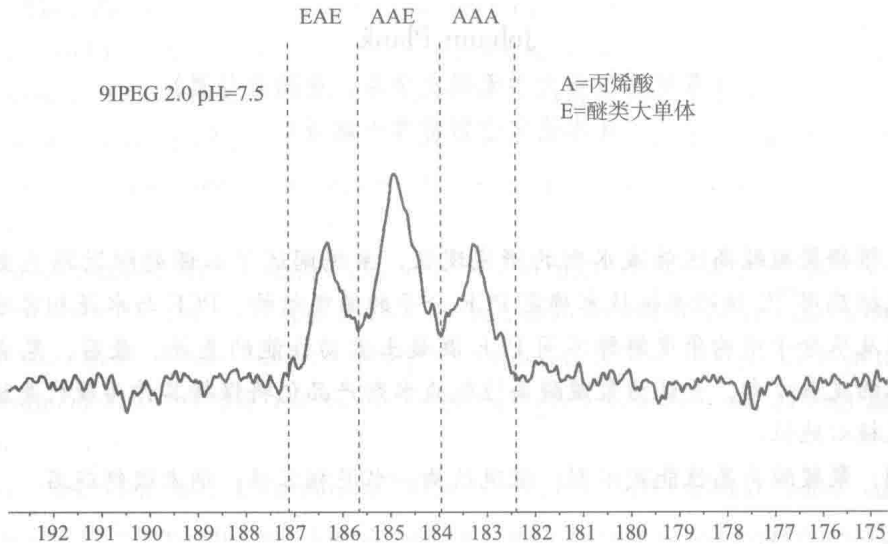


图1 9IPEG 2.0 PCE 高性能减水剂的¹³C 核磁共振谱图

3 PCE 与水泥的适应性

施工过程中, 由于与水泥适应性差而导致聚羧酸高性能减水剂性能降低的案例屡见不鲜, 对于某些特殊的水泥品种, PCE 甚至完全丧失工作性。显然, 这类水泥的组分在一定程度上阻碍了 PCE 的分散作用。

本课题组最近研究了超过 20 种具有不同组分的水泥样品, 其中 C₃A 含量浮动范围为 1.0% ~ 9.8% (质量分数)。对于这 20 种不同的水泥样品, 应用同一种 MPEG 型聚羧酸高性能减水剂达到相同的流动度所使用的掺量相差很大 (见表 1)。根据净浆试验结果, 选择两种“困难”水泥 (CEM I 32.5 R 和 42.5 R) 做进一步研究。结果发现, 在两种情况下 PCE 与水泥的适应性问题较易出现, 一种情况是水泥中 C₃A 含量较高 (≥6%), 另一种情况是 PCE 影响了水泥早期水化产物—钙矾石晶体的尺寸, 使得其尺寸由常规的 2 μm 缩小至仅有几百纳米 (图 2)。

表 1 不同 PCE 样品的饱和掺量 (W/C=0.30) %

样品	MPEG-7	MPEG-25	APEG-34	APEG-34AM	IPEG-25
CEM I 32.5 N	0.25	0.11	0.21	0.10	0.10
CEM I 32.5 R	>1.00	>1.00	>1.00	0.25	0.51
CEM I 42.5 N	0.35	0.16	0.29	0.14	0.13