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**RECENT ADVANCES IN
CONCRETE
TECHNOLOGY**

**PROGRÈS RÉCENTS DANS
LE DOMAINE DE LA
TECHNOLOGIE DU BÉTON**

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Fifth CANMET/ACI International Conference on Recent Advances in Concrete Technology

Editor

V. M. Malhotra



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PREFACE

The Canada Centre for Mineral and Energy Technology (CANMET) of Natural Resources Canada, Ottawa, has played a significant role in Canada for over 35 years in the broad area of concrete technology. In recent years, CANMET has become involved increasingly in research and development dealing with the supplementary cementing materials, high-performance normal-weight and lightweight concretes and alkali-aggregate reactions. As part of CANMET's technology transfer program, an international symposium on Advances in Concrete Technology was sponsored jointly with the American Concrete Institute and other organizations, and held in Athens in May 1992. In June 1995, CANMET, in association with the American Concrete Institute and other organizations in Canada and the USA sponsored the Second CANMET/ACI International Symposium on Advances in Concrete Technology in Las Vegas, USA. For the Athens symposium, the CANMET publication "Advances in Concrete Technology" constituted the proceedings of the symposium; for the Las Vegas symposium, the proceedings were published by the American Concrete Institute as ACI SP-154.

In August 1997, CANMET, in association with the American Concrete Institute and other organizations in Canada and New Zealand, sponsored the Third CANMET/ACI International Symposium on Advances in Concrete Technology in Auckland, New Zealand. The main purpose of the symposium was to bring together representatives from industry, universities, and government agencies to present the latest information in the subject area of the symposium, and to explore new areas of needed research and development. Thirty-three refereed papers from 15 countries were presented. In addition to the refereed papers, more than 20 other papers were presented and distributed at the symposium. The proceedings were published as ACI SP-171.

In June 1998, CANMET, in association with the American Concrete Institute, Japan Concrete Institute, and several other organizations in Canada and Japan, sponsored the Fourth CANMET/ACI International Conference on Recent Advances in Concrete Technology in Tokushima, Japan. More than 80 papers from 20 countries were received and reviewed in accordance with the policies of the American Concrete Institute. Sixty-one refereed papers were accepted for presentation at the conference and for publication in ACI SP-179. In addition to the refereed papers, more than 30 other papers were presented and distributed at the symposium.

In July-August 2001, CANMET, in association with the American Concrete Institute and several organizations in Singapore, sponsored the Fifth

CANMET/ACI International Conference on Recent Advances in Concrete Technology in Singapore. More than 100 papers from more than 25 countries were received and reviewed in accordance with the policies of the American Concrete Institute. Forty-six refereed papers were accepted for presentation at the conference and are published here as ACI SP-200. In addition to the refereed papers, more than 25 other papers were presented and distributed at the conference.

Thanks are extended to the members of the CANMET/ACI paper review panel which met in Torremolinos, Spain, in June 2000 to review the papers. Without their prompt review and constructive comments, it would not have been possible to bring out the ACI special publication for distribution at the symposium in Singapore. The cooperation of the authors in accepting reviewers suggestions, and in revising their manuscripts accordingly is greatly appreciated. Particular thanks are extended to H. S. Wilson, Consultant, Ottawa, and G. D. Brearley and M. Venturino, both of CANMET, for their help in the processing of the draft manuscripts.

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R. Hartford, Manager, Editorial & Production, and her staff at ACI deserve a very special mention for their assistance in getting this publication ready on time for distribution in Singapore.

V. M. Malhotra, P.Eng.

Editor

Chairman, CANMET/ACI Fifth International Conference
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Recycling of Demolished Concrete

by S. Nagataki and K. Iida

Synopsis: In Japan, it has been predicted that enormous quantities of demolished concrete will be produced in the future. Therefore, a great deal of research is being conducted to find ways to recycle this demolished concrete as concrete aggregate. However, because the characteristics of the original concrete are not well known in many cases, the results are also not clear. Moreover, the recycling of the cement in concrete is also necessary from the standpoint of resolving global environmental problems and achieving sustainable development. This paper describes the properties of the concrete made with recycled aggregates from the original concrete of known quality and the recycling of the cement in concrete as the approach that should be taken in the 21st century.

Keywords: aggregate; carbon dioxide emissions; cement; concrete; recycling

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INTRODUCTION

The concept of sustainable development has been proposed for what has been called our “finite deteriorating planet”. This concept involves reducing the environmental burden to the greatest extent possible and building a society based on recycling.

In the concrete industry as well, recycling is currently being studied for demolished concrete. This material is already being reused as road base course and foundation material. In the future, however, this demolished concrete must be recycled as concrete aggregate, and recycling must also be extended to include the cement in the concrete as well.

CONCRETE RECYCLING IN JAPAN

According to the Ministry of Welfare, 400 million tons of industrial wastes are produced in Japan each year (Fig. 1). The construction industry is a major source of these wastes. The industries are currently promoting waste reduction and recycling, and so the quantity of final disposal wastes now represents about 17% of the total. However, the capacity of final disposal sites is currently limited to only three years.

A survey conducted by the Ministry of Construction in 1995 found that

the total quantity of construction wastes produced each year came to nearly 100 million tons, and demolished concrete was the leading component of these wastes, accounting for 37 million tons (Fig. 2). While 65% of concrete wastes are recycled, they are reused mainly as road base course and foundation material, and almost none is recycled back into concrete.

Almost all of the concrete structures in Japan have been built over the last 50 years. As a result, the quantity of demolished and discarded concrete is expected to increase dramatically in the coming years (Fig. 3) (1). For this reason, there are many ongoing researches aiming to recycle waste concrete as concrete aggregate. However, in many cases the characteristics of the original concrete are not well known, so that the properties of the concrete made with recycled aggregates are not predictable.

Furthermore, even recycling of demolished concrete as concrete aggregate becomes possible, it would not be adequate for a closed recycling system to turn old concrete back into new concrete. Accordingly, special types of fully recyclable concrete that use limestone aggregate (2) and recycling of the cement in demolished concrete as well (3) have been proposed.

The objectives of the study are to clear the properties of recycled aggregate concrete and to propose new recycling system that covers cement as well.

RECYCLED CONCRETE AS AGGREGATE

Original Concrete

The original concrete prepared for this investigation contained normal portland cement, river sand, crushed stone and an air-entraining and water-reducing admixture. The properties of the original aggregates are shown in Table 1. Original concrete cubes for producing recycled aggregate, 300 mm by 300 mm by 300 mm in size, were made with three mixture proportions representing high, moderate, and low strength as shown in Table 2. After water curing, the original concrete cubes were demolded at the age of 28 days and exposed outdoors. They were processed into recycled aggregate at the ages of 1 month, 1 year, and 2 years.

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At the same time, cylindrical specimens 100 mm in diameter and 200 mm in length were fabricated. The properties of the original concretes are shown in Table 3. The compressive strength of concrete specimen at 28 days under standard curing conditions were 60.7 MPa for HSC, 49.0 MPa for MSC, and 28.3 MPa for LSC, respectively.

Production of Recycled Aggregate

The crushing was carried out in accordance with the flow chart shown in Fig.4. The original concrete underwent primary crushing with a jaw crusher and secondary crushing with an impact crusher. This stage is referred to as crushing level 1. Further crushing stages using an improved jaw crusher with a strong grinding effect once and twice are referred to as crushing level 2 and crushing level 3, respectively. The recovery ratios of coarse aggregate for crushing level 1, 2, and 3 were 60%, 45%, and 30%, respectively, with the mass of the original concrete being 100%, in which the mass ratio of original coarse aggregate was approximately 43%. The strength of the original concrete had no appreciable effects on these recovery ratios. The properties of the recycled coarse aggregates produced are given in Table 5. Generally speaking, using higher strength concrete and a higher crushing level can produce recycled coarse aggregate of better quality. However, the aggregates did not satisfy the Japanese Industrial Standard (JIS) for concrete aggregate in terms of density and water absorption.

Strength Characteristics

The materials used with recycled coarse aggregate comprised normal portland cement, river sand, and an air-entraining and water-reducing admixture. The river sand was the same as used in the original concrete. The mixture proportions are given in Table 4. When the water-binder ratio was 0.35 or less, silica fume and an air-entraining and high-range water-reducing admixture were used.

Fig. 5 shows the relationship between the binder-water ratio and the 28-day compressive strength of recycled concrete using aggregates reclaimed at 1 month

with 3 levels of crushing. The compressive strength of concrete containing recycled coarse aggregate depends on the compressive strength of the original concrete. The compressive strength of recycled aggregate concrete was similar to original aggregate concrete and retained a linear relationship with the binder-water ratio of up to 1.5 times the 28-day strength of the original concrete. The level of crushing had no appreciable effect on the compressive strength.

Fig. 6 shows the relationship between the binder-water ratio and the 28-day compressive strength of recycled concrete using aggregates reclaimed at 1 month, 1 year, and 2 years with crushing level 2. The age of concrete at the time of crushing (crushing age) had no appreciable influence on the compressive strength of recycled concrete using aggregate made from the original concrete with high strength (HSC) and moderate strength (MSC). For the original concrete with low strength (LSC), the compressive strength of recycled concrete was somewhat influenced by crushing age. The concrete made with the recycled aggregate reclaimed at 1 year or 2 years exhibited higher compressive strength in comparison to the recycled aggregate reclaimed at 1 month.

Resistance to Freezing and Thawing

The materials used were the same as those used in the test of concrete strength. The mixtures were proportioned to provide a slump of 8 ± 2.5 cm and air content of $4.5 \pm 1.5\%$, with the water-cement ratio being constant at 0.55. The freezing and thawing test procedure was in accordance with ASTM C 666 Procedure A, though the test was initiated at an age of 28 days.

Fig. 7 shows the changes in the relative dynamic modulus with freezing and thawing cycles for the recycled concrete using aggregates reclaimed at 1 month with 3 levels of crushing. Recycled aggregate concrete showed larger losses in the relative dynamic modulus than the original aggregate concrete. The losses were larger for aggregates made from low and moderate strength concrete. Large losses were also found in the case where the crushing level was low. Though the resistance to freezing and thawing decreased, the durability factor was greater than 70 at 300 cycles, which is generally considered as satisfactory.

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Fig. 8 shows the changes in the relative dynamic modulus with freezing and thawing cycles for the recycled concrete using aggregates reclaimed at 1 month and 1 year with crushing level 1. At crushing age of 1 year, the resistance to freezing and thawing of recycled aggregate concrete was improved and similar to original aggregate concrete.

Fig. 9 shows the relationship between the mass losses by the soundness test (5) on coarse aggregate and the durability factor of the concrete using these aggregates at 300 cycles. For crushing age of 1 month, the mass losses correlated relatively well with the durability factor. Though the threshold value for mass losses is generally assumed to be 12%, a mass loss of as high as 60% of recycled coarse aggregate provided sufficient resistance to freezing and thawing for the concrete. For crushing age of 1 year, the durability factor was retained over 90 regardless of the soundness of recycled coarse aggregate.

Fig. 10 shows the changes in the relative dynamic modulus with freezing and thawing cycles for the recycled concrete using aggregates from non-AE concrete. All of the concrete were poor in resistance to freezing and thawing. Increasing the air content or lowering the water-cement ratio in recycled aggregate concrete did not improve the resistance sufficiently.

SUSTAINABLE DEVELOPMENT

Concept

Of all the processes in the construction industry, cement manufacture releases the largest quantities of carbon dioxide. Also, to make concrete a sustainable material, the cement in concrete must be recycled. There has been some progress in recycling aggregate from demolished concrete. However, this paper proposes a change in this approach, to place primary emphasis on recycling the cement materials from demolished concrete -- in other words, to aim to reclaim the fine powder containing as much cement components as possible. This would result in the recycling of good quality aggregate as well.

Reclaimed Cement

Table 6 shows the chemical composition of normal portland cement and aggregate powder. The chemical composition of aggregate powder was estimated from the chemical composition of powders produced by several aggregate recycling factories (1). Table 7 shows the chemical composition of recycled powder for different aggregate ratios in the recycled powder. They are calculated from Table 6.

One method of reclaiming aggregate from concrete is to embrittle the paste components by a thermal treatment (6). After heating concrete rubble up to 300°C, the separation of cement matrix from aggregate by ball mill is tested. According to the reports (7), the recycled aggregate crushed after the thermal treatment fulfills the Japanese Industrial Standard (4) for concrete aggregate, for both coarse aggregate and fine aggregate, and the coarse aggregate is almost completely unbroken. The aggregate ratio of the powder is estimated at 35%. This is the result of grinding the aggregate, and so future studies will be conducted on the assumption that the aggregate ratio can be reduced to 30%.

Table 8 shows the results of how the quantity of limestone can be reduced when the recycled powder with an aggregate ratio of 30% shown in Table 7 is used as clinker raw material. The value for $\text{Na}_2\text{O}_{\text{eq}}$ slightly exceeds the standard for normal portland cement, but in principle recycling of powder to make cement is thought to be possible.

Evaluation of Recycling

Under the assumption that the recycled powder above can be recycled to make cement, the resources, energy and carbon dioxide emissions for a material flow of 1 m³ of concrete are evaluated. The cement was the aforementioned normal portland cement and recycled portland cement, and the conditions were those shown below. Carbon dioxide emissions were expressed by the commonly used measure of carbon mass (kg-C).

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[Cement manufacture]

Resources: Mining loss for limestone, clay and silica = 0%

Energy: For mining and pulverizing limestone and silica = 42 kJ/kg (8)

For calcination = 2954 kJ/kg of clinker (9)

= 1882 kJ/kg of clinker raw material

In calculating the energy required to calcine recycled cement, in order to reflect the loss of decarboxylation energy and the loss of clinker raw materials, the firing energy was converted to the value per unit of clinker raw materials, as shown above.

Carbon dioxide emissions:

From limestone = 0.12 kg-C/kg (9)

From calcination = 0.0626 kg-C/kg of clinker (9)

From distribution = 0.0025 kg-C/kg of cement (10)

[Aggregate production]

Aggregates are crushed stone and crushed sand. The conditions for each are shown below.

Resources: Mining loss for crushed stone and crushed sand = 30%

Pulverization loss for crushed stone and crushed sand = 10%

Energy: For manufacture of crushed stone and crushed sand = 42 kJ/kg (8)

Carbon dioxide emissions:

From manufacture of crushed stone and crushed sand = 0.00189 kg-C/kg (10)

From distribution of crushed stone and crushed sand = 0.00056 kg-C/kg (10)

[Concrete demolition and thermal treatment]

Under the assumption that the demolished concrete is transported to a place near the factory and then fired to 300°C and pulverized, the conditions for the demolition and heating processes are shown below.

Energy: For heating process = $0.8 \text{ kJ/kg}^\circ\text{C} \times 300^\circ\text{C} \times 1.25$

= 300 kJ/kg of concrete

For demolition, transport and pulverizing = 42 kJ/kg (8)

Carbon dioxide emissions:

From heating process = 0.0161 kg-C/MJ (11)

From pulverizing = 0.00189 kg-C/kg of concrete (10)

From transport = 0.00056 kg-C/kg of concrete (10)

[Recycled concrete]

Table 9 shows the concrete mixture proportions.

Fig. 11 shows the material flow, energy and carbon dioxide emissions per 1 m³ of concrete studied under the conditions shown above.

First, the use of new materials and the use of recycled materials were compared in terms of resources. Table 10 shows the consumption of resources for the major components. When the cement in concrete is recycled, the amount of limestone mined decreases to 61% compared with when new materials are used. Clay and silica are not needed; the quantity of iron slag consumed drops to 58%, and the quantity of stone for aggregate drops to 9%. Moreover, the quantity of stone for aggregate that is discarded is reduced from 774 kg to 71 kg (Fig. 11). When cement is recycled, of the 474 kg of recycled powder, 287 kg of powder is left over. This is small in comparison to the quantity of new stone for aggregate that is discarded, and it indicates that in terms of quantity the refining of recycled powder is possible. In this way, recycling of the cement in concrete will make it possible to both conserve resources and reduce the destruction of the natural environment caused by the mining of resources.

Table 11 also shows a comparison of energy requirements. When cement is recycled, the demolished concrete is heated, and so energy requirements are 58% greater than when new materials are used. In this study, it was assumed that concrete would be recycled at a cement factory, and so some reduction of energy requirements can be anticipated though the use of waste heat. More radically, improvements in cement calcinating processes through the use of different raw materials are also anticipated. However, in the current situation, an increase in energy requirements is judged to be unavoidable.

Table 12 shows a comparison of carbon dioxide emissions. When cement is recycled, carbon dioxide emissions from cement manufacture are reduced by 29%. Even if the increase in carbon dioxide emissions due to the heating process is factored in, this still represents an overall decrease of 11%. The problem of carbon dioxide emissions is a serious one for the global environment. At the Kyoto conference on global warming held in 1997 (Third Conference of the Parties to the United Nations Framework Convention on Climate Change), Japan's target for

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reduction of CO₂ emissions was 6%. Reducing carbon dioxide emissions due to cement manufacture, in which large quantities of carbon dioxide fixed in limestone are discharged, would have tremendous significance.

As we have seen, a closed recycling system of concrete recycling, in which cement and aggregate are recycled from demolished concrete, would increase energy requirements somewhat, but it would also conserve resources, reduce the destruction of the natural environment due to the mining of these resources and reduce carbon dioxide emissions. Even recycling concrete aggregate alone would enable conservation of resources and reduce the destruction of the natural environment that results from the mining of these resources. However, it would not reduce carbon dioxide emissions, and so this would be only a slight improvement over the current reuse of this aggregate as road base coarse and foundation material. Surely concrete recycling that includes cement and is aimed at reducing the amount of limestone used to make cement is the approach that recycling should take in the 21st century.

SUMMARY

- (1) In recycling demolished concrete, change is necessary from reuse as a road foundation material to recycling for concrete aggregate. Furthermore, recycling should include cement.
- (2) The recycled aggregate does not satisfy the Japanese Industrial Standard requirements for concrete aggregate.
- (3) The quality of recycled concrete as aggregate depends on the quality of original concrete. The recycled aggregate was suitable for concrete within the strength level of original concrete without special further treatment. The resistance to freezing and thawing of recycled concrete was not satisfactory unless the original concrete was air-entrained concrete. The amount of mortar adhered to the recycled aggregate had little effect on the strength and durability of recycled concrete.
- (4) For reducing carbon dioxide emissions that causes global warming, and for making concrete sustainable, it is desirable that concrete recycling includes cement as well.

(5) The possibility of reclaiming cement with reduced limestone was studied in the process of concrete crushing after heating. The results show that concrete recycling included cement needs more energy, but uses less resources and discharges less carbon dioxide.

(6) For making concrete friendly to the global environment and sustainable in the 21st century, cement reclaiming should be the priority in the recycling of demolished concrete.

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Table 1. Properties of the original fine and coarse aggregate

Aggregate	Density (g/cm ³)		Absorption (%)	Under 75 μ m (%)	Soundness (%) *	Bulk density (kg/m ³)	Solid Vol. (%)	F.M
	Oven Dry	S.S.D						
Fine	2.55	2.62	2.59	1.64	2.6	1570	66.6	2.87
Coarse	2.62	2.65	0.94	0.05	9.1	1700	59.8	6.48

* by JIS A 1122: Method of test for soundness of aggregates by use of sodium sulfate

Table 2. Mixture proportions of the original concretes

Type	Size* (mm)	Slump (cm)	Air (%)	W/C	s/a (%)	Unit content (kg/m ³)					
						Water	Cement	F.Agg	C.Agg	AEWR	AEHRWR
HSC	20	8±2.5	4.5±1.5	0.35	41	170	486	679	986	-	4.37
MSC	20	8±2.5	4.5±1.5	0.45	43	170	378	749	1004	-	3.40
LSC	20	8±2.5	4.5±1.5	0.625	47	167	267	867	988	0.668	-

* maximum size of aggregate

Table 3. Properties of the original concretes

Type	Slump (cm)	Air (%)	Compressive strength (MPa)						Young's modulus (GPa)	
			Standard curing		Seal-curing on site				Seal-curing on site	
			7 days	28 days	7 days	1 month	1 year	2 years	1 month	1 year
HSC	20*	4.4	48.8	60.7	47.1	56.1	60.1	62.3	28.5	32.1
MSC	10*	4.0	37.9	49.0	33.8	40.5	41.7	44.3	26.2	30.0
LSC	7.5	4.5	19.3	28.3	18.1	24.4	25.5	27.0	22.2	24.5

* superplasticized

Table 4. Mixture proportions for strength test

Size* (mm)	Slump (cm)	Air (%)	W/ (C+SF)	s/a (%)	Unit content (kg/m ³)						
					Water	Cement	Silica Fume	Fine Agg.	Coarse Aggregate	AEWR	AEHRWR
20	8 ± 2.5	4.5 ± 1.5	0.25	32.1	160	576	64	496	m ³ /m ³ 0.4	-	4.48
			0.30	36.0		480	53	588		-	3.73
			0.35	38.4		411	46	654		-	2.29
			0.40	41.0		400	-	715		1.25	-
			0.45	42.0		356	-	752		1.11	-
			0.55	43.5		291	-	806		0.91	-
			0.65	45.0		246	-	843		0.77	-

* maximum size of aggregate

Table 5. Properties of recycled coarse aggregate made from concrete

Original Concrete	Crush Level	Crush Age (year)	Adhered Mortar* (%)	Density (g/cm ³)		Absorption (%)	Under 75 μ m (%)	Soundness** (%)	Bulk density (kg/m ³)	Solid volume (%)	F.M
				O.D	S.S.D						
Original aggregate			0	2.62	2.65	0.94	0.05	9.1	1700	59.8	6.48
HSC	1	0.1	39.0	2.31	2.43	5.03	0.04	34.1	1390	60.2	6.65
		1	52.3	2.31	2.42	4.88	0.22	29.7	1360	58.9	6.67
		2	49.8	2.33	2.43	4.31	0.10	14.1	1400	60.1	6.58
	2	0.1	26.0	2.37	2.47	4.38	0.03	24.9	1480	62.4	6.61
		1	42.2	2.36	2.46	4.12	0.22	15.6	1460	61.8	6.55
		2	40.1	2.38	2.47	3.58	0.10	11.3	1460	61.2	6.50
	3	0.1	22.2	2.44	2.52	3.40	0.04	22.8	1530	62.7	6.66
		1	30.2	2.43	2.51	3.14	0.30	8.1	1490	61.2	6.51
		2	35.8	2.43	2.50	3.01	0.14	5.4	1520	62.6	6.60
MSC	1	0.1	39.3	2.28	2.42	5.96	0.02	57.0	1390	61.0	6.67
		1	55.1	2.28	2.41	5.58	0.17	48.3	1370	60.0	6.57
		2	51.3	2.29	2.41	5.13	0.11	19.6	1380	60.2	6.62
	2	0.1	30.2	2.34	2.46	4.96	0.04	52.9	1460	62.4	6.63
		1	46.4	2.34	2.45	4.64	0.24	22.8	1420	60.6	6.51
		2	44.6	2.33	2.44	4.50	0.12	20.8	1460	62.5	6.69
	3	0.1	23.8	2.39	2.49	4.30	0.10	34.7	1500	62.8	6.58
		1	32.4	2.42	2.50	3.19	0.41	18.4	1490	61.5	6.39
		2	21.0	2.47	2.54	2.68	0.04	9.5	1520	61.4	6.45
LSC	1	0.1	39.1	2.26	2.40	5.95	0.07	52.0	1360	60.2	6.62
		1	52.2	2.23	2.37	6.27	0.17	49.1	1340	60.1	6.59
		2	51.4	2.29	2.41	5.28	0.04	40.2	1360	59.4	6.53
	2	0.1	30.1	2.31	2.43	5.08	0.13	44.3	1440	62.3	6.60
		1	45.4	2.28	2.40	5.42	0.21	36.5	1400	61.5	6.67
		2	35.0	2.35	2.45	4.36	0.08	20.8	1420	60.5	6.46
	3	0.1	19.2	2.37	2.47	4.35	0.10	35.3	1480	62.4	6.66
		1	32.3	2.39	2.48	3.76	0.14	22.5	1510	63.2	6.69
		2	28.5	2.42	2.50	3.43	0.04	18.0	1510	62.5	6.61

* determined by the hydrochloric acid dissolution method

** by JIS A 1122: Method of test for soundness of aggregates by use of sodium sulfate

Table 6. Chemical composition of cement and aggregate powder (%)

	Ig. loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ Oeq	Cl
Normal portland cement	0.7	22.2	5.2	3.1	64.8	1.5	1.9	0.70	0.005
Aggregate powder	8.6	68.6	13.4	3.9	0.0	2.0	0.0	3.35	0.077

Table 7. Chemical composition of recycled powder (%)

Aggregate ratio (%)	Ig. loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ Oeq	Cl
20	2.2	31.5	6.8	3.3	51.8	1.6	1.5	1.23	0.019
30	3.0	36.1	7.7	3.3	45.4	1.7	1.3	1.50	0.027
40	3.8	40.8	8.5	3.4	38.9	1.7	1.1	1.76	0.034
50	4.6	45.4	9.3	3.5	32.4	1.8	0.9	2.03	0.041

Table 8. Normal portland cement made with recycled powder

		Chemical composition (%)								Quantity (kg)
		Ig. loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ Oeq	
Clinker	Limestone	43.0	1.1	0.3	0.3	54.2	0.5	0.0	0.03	668
	Recycled powder	3.0	36.1	7.7	3.3	45.4	1.7	1.3	1.50	622
	Iron slag	9.1	16.4	5.3	54.4	9.4	1.5	1.7	0.53	15
	Total	23.5	17.7	3.8	2.3	49.2	1.1	0.6	0.73	1325
Cement	Clinker	0.0	23.4	5.1	3.1	65.2	1.4	0.8	0.96	1000
	Gypsum	(20.9)				32.6		46.5		32
	Cement*	0.7	22.7	4.9	3.0	64.1	1.4	2.3	0.93	1000
JIS R 5210 N.P.C		<3.0					<5.0	<3.0	<0.75	<0.02

* HM=2.05, SM=2.88, IM=1.66

Table 9. Mixture proportions for evaluation of concrete recycling

Size* (mm)	Slump (cm)	Air (%)	W/C	s/a (%)	Unit content (kg/m ³)				
					Water	Cement	Fine Agg.	Coarse Agg.	AEWR
20	12.0	4.0	0.55	44	170	310	785	1015	

* maximum size of aggregate

Table 10. Consumption of resources (kg/concrete m³)

	Limestone	Clay	Silica	Iron slag	Stone for agg.
Using of new materials	342 (100%)	73 (100%)	10 (100%)	8.6 (100%)	2574 (100%)
Recycling cement and aggregate	207 (61%)	0 (0%)	0 (0%)	5.0 (58%)	235 (9%)

Table 11. Energy requirements (MJ/concrete m³)

	Cement production	Agg. production	Recycling	Total
Using of new materials	901 (100%)	76 (100%)	0	977 (100%)
Recycling cement and aggregate	757 (84%)	7 (9%)	780	1544 (158%)

Table 12. Carbon dioxide emissions (kg-C/concrete m³)

	Cement production	Agg. production	Recycling	Total
Using of new materials	71.5 (100%)	4.4 (100%)	0	75.9 (100%)
Recycling cement and aggregate	50.6 (71%)	0.4 (9%)	16.6	67.6 (89%)

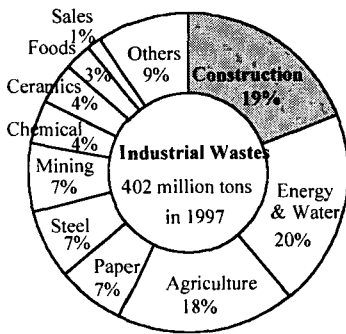


Fig. 1. Industrial wastes in Japan

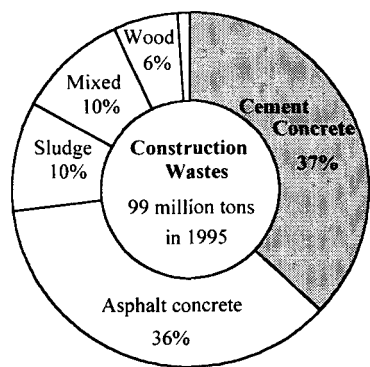


Fig. 2. Construction wastes in Japan

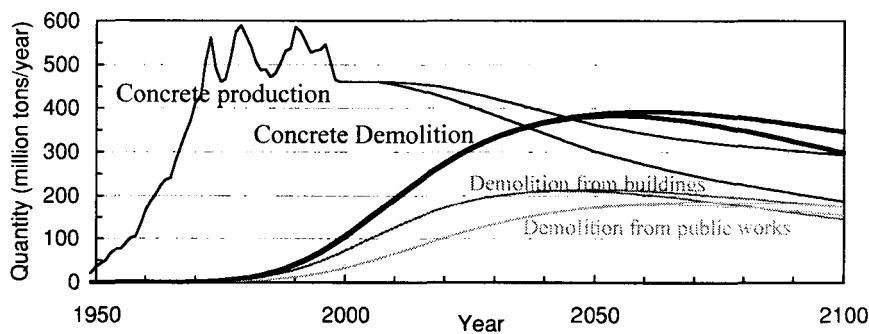


Fig. 3. Estimated Concrete Production and Demolition in Japan

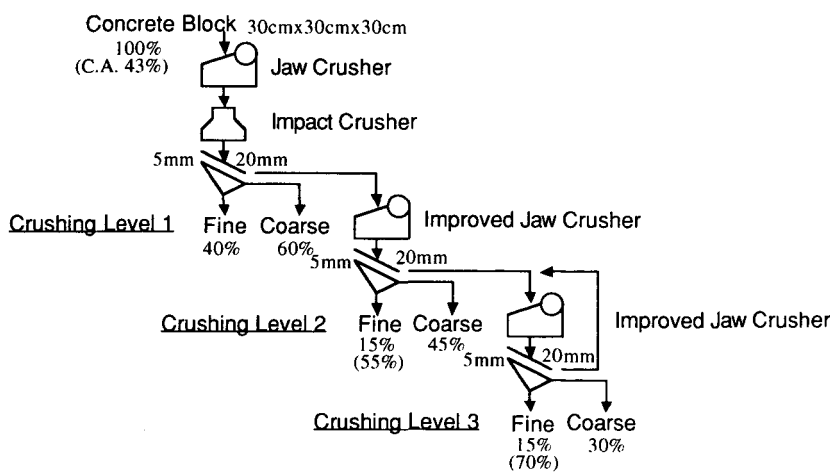


Fig. 4. Production flow for recycling aggregate

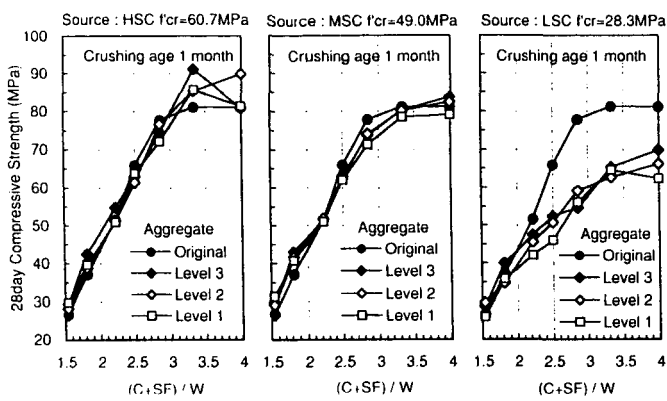


Fig. 5. Compressive strength of recycled concrete for different crushing levels

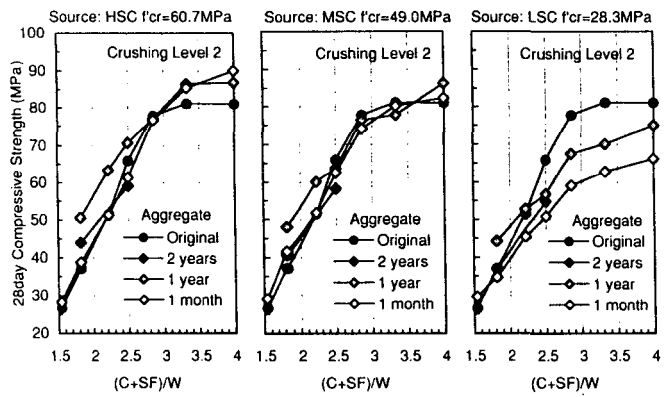


Fig. 6. Compressive strength of recycled concrete for different crushing ages

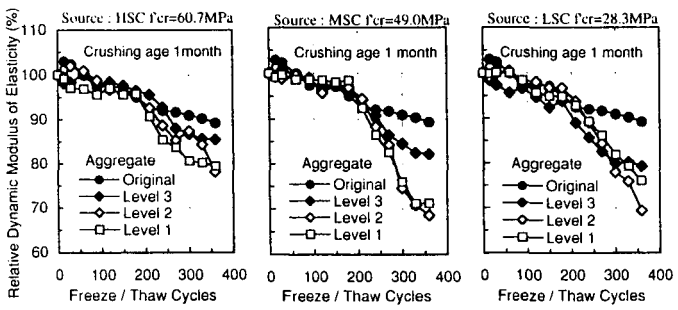


Fig. 7. Resistance to freeze and thawing of recycled concrete for different crushing levels

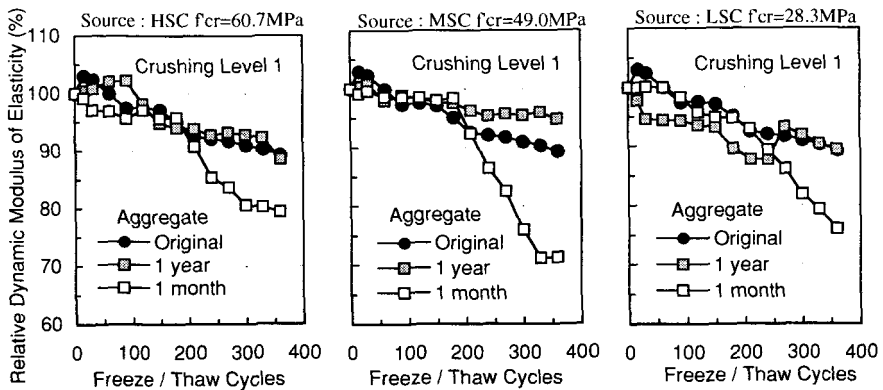


Fig. 8. Resistance to freeze and thawing of recycled concrete for different crushing ages

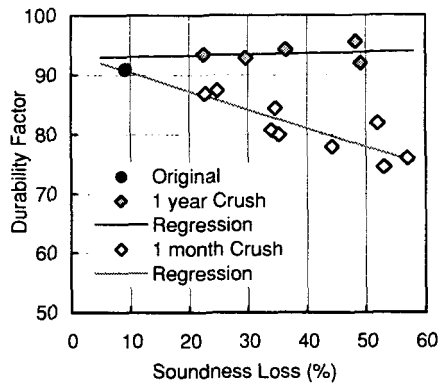


Fig. 9. Durability factor of concrete and soundness loss of aggregates as tested by JIS A 1122

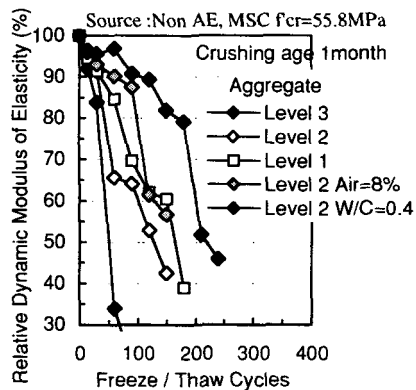


Fig.10. Resistance to freeze and thawing of recycled concrete as aggregate from non-AE concrete

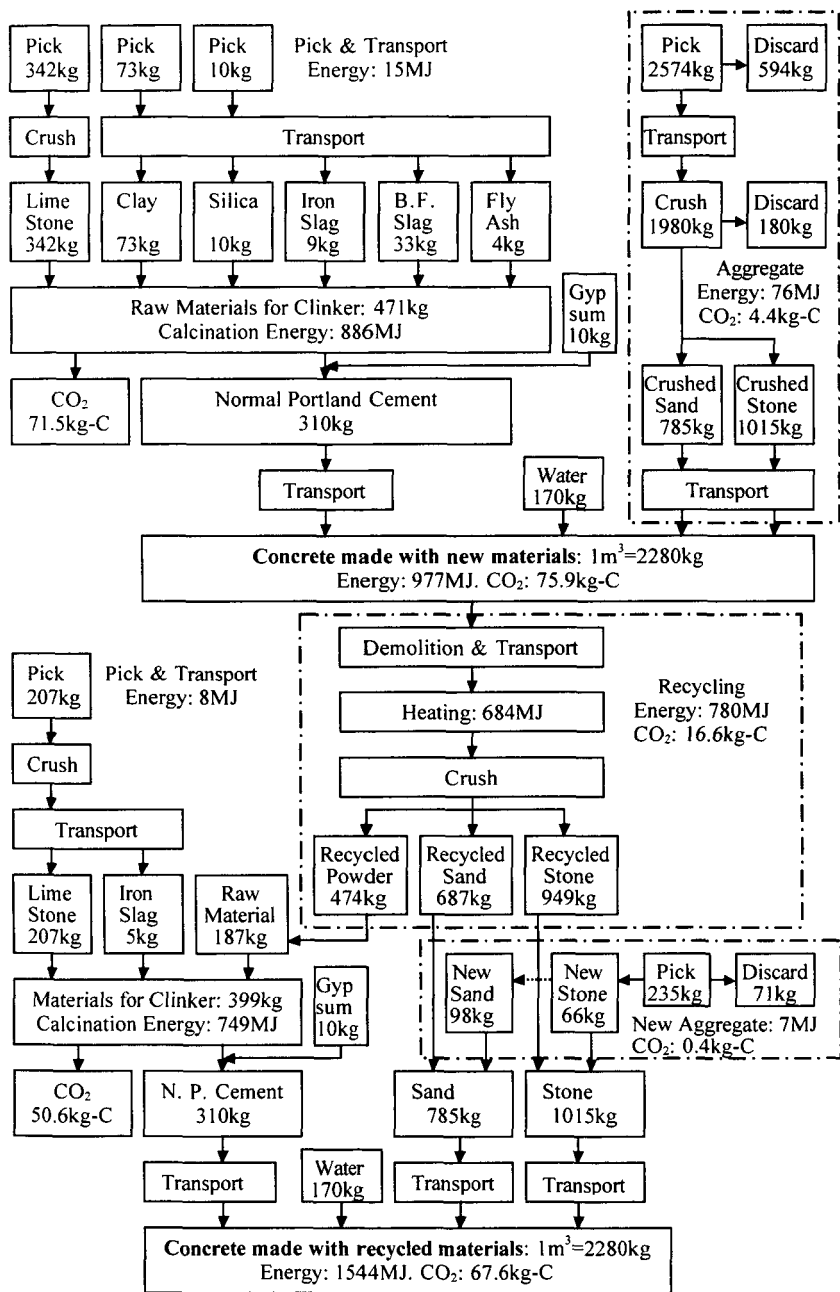


Fig. 11. Material flow for 1 m³ concrete with energy consumption and carbon dioxide emissions

Ettringite Formation and Sulfate Attack on Concrete

by M. Collepardi

Synopsis: The paper presents a critical review of the relationship between ettringite formation and sulfate attack. Ettringite formation is associated with expansion. However, not necessarily any ettringite-related expansion is related to sulfate attack.

Early ettringite formation (*EEF*) which occurs *immediately* (within hours) in a plastic fresh mixture does not produce any damaging expansion and is associated with the regulation of setting time of portland cement paste. Expansion after the hardening of cement paste can be advantageously used for development of chemical prestress in expansive cements.

Delayed ettringite formation (*DEF*) occurs at *late* ages and the related *heterogeneous* expansion in a very rigid hardened concrete can produce cracking and spalling. Two different types of *DEF* are examined depending on the sulfate source: *DEF* caused by external sulfate attack (*ESA*) or internal sulfate attack (*ISA*).

ESA, related to the interaction of environmental sulfate with the cement matrix, can be precluded by the use of impermeable concrete.

On the other hand, *ISA* occurs in a sulfate-free environment due to the interaction of internal sulfate (from cement or gypsum contaminated aggregate) with calcium-aluminate hydrates of the cement paste.

Two different mechanisms of *DEF* caused by *ISA* are examined. The first one is based on the thermal decomposition of ettringite in high-temperature cured concrete elements and the subsequent re-formation of ettringite at ambient temperature in a saturated atmosphere. According to the second mechanism *ISA* is based on a chain of three essential events (*microcracking*, *late sulfate release*, and *exposure to water*) and *DEF* could occur even at room temperature.

Keywords: concrete; delayed ettringite formation; ettringite; sulfate attack

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INTRODUCTION

Ettringite formation is considered to be the cause of most of the expansion and disruption of concrete structures involved in the sulfate attack (1). However, not necessarily any sulfate attack is caused by ettringite formation (2). Moreover ettringite formation can be advantageously used without any sulfate attack on concrete.

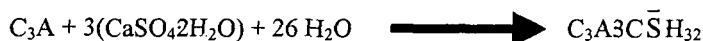
The purpose of this review is to present critical analysis of the relationship between ettringite formation and sulfate attack including some special cases of this attack such as those related to thaumasite formation or "delayed ettringite formation" as intended according to the terminology currently used (3,4).

The term "sulfate attack", as used here, means deterioration of concrete involving any type of sulfate interactions with cement paste independently of the curing temperature and sulfate source. In other words, both concrete structures cured at ordinary temperatures and those steam-cured will be examined irrespective of whether the source of sulfate is external or internal. This paper does not cover damage which is not specifically attributed to the chemical sulfate attack such as crystallization of water-soluble sulfate salts (5).

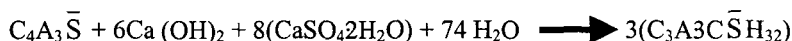
ETTRINGITE FORMATION

Ettringite formation is associated with expansion and many hypotheses of ettringite related expansion have been advanced (2-4). However, not necessarily any ettringite-related expansion produces damaging disruption of concrete structures.

When ettringite occurs **homogeneously** and **immediately** (within hours), it does not cause any significant localized disruptive action (*early ettringite formation, EEF*). This type of harmless ettringite formation happens, for instance, when gypsum reacts with anhydrous calcium aluminate in a through-solution reaction and acts as a set retarder in portland cement mixtures (Table 1):



The retardation has been attributed to the early formation of an ettringite layer or membrane over the surfaces of the cement grains soon after mixing (3). This type of ettringite may also be called "normal" ettringite as opposite to "secondary" ettringite which will be defined later. The formation of "normal" ettringite occurs in a plastic concrete and does not produce any significant stress. Another example of harmless and useful *EEF* is Type K expansive cement containing a calcium aluminate sulfate ($C_4A_3\bar{S}$) which, under proper restraint, hydrates within a few days producing uniformly distributed ettringite and homogeneous expansion throughout the hardened concrete:



In such a case, the restrained expansion is advantageously transformed into a rather useful stress (0.2 - 0.7 MPa in shrinkage-compensated concrete and 3 to 8 MPa in self-stressed reinforced concretes) provided that the concrete has reached a certain strength level and a corresponding modulus of elasticity.

On the other hand, when ettringite forms **later** (after several months or years) - *delayed ettringite formation (DEF)* - the related **heterogeneous** expansion in a very rigid hardened concrete can produce cracking and spalling. The disruptive effect is due to the non-uniform expansion localized only in the area of the concrete structure where ettringite forms. Therefore *DEF*, and not *EEF*, is associated with a damaging sulfate attack (Table 2). There are two different types of *DEF*-related damage depending on the sulfate source (Table 3): external or internal sulfate attack. External sulfate attack (*ESA*) occurs when environmental sulfate (from water or soil) penetrates concrete structures. Internal sulfate attack (*ISA*) occurs in a sulfate-free environment when the sulfate source is inside the concrete and comes from either cement with a high sulfate content or from aggregates contaminated with gypsum.

According to the terminology currently used (3,4), "*DEF*" or "secondary ettringite" (6) is related only to the internal sulfate attack particularly in heat-cured concrete structures where "normal" ettringite, responsible for setting regulation, is thermally decomposed and then forms again in an atmosphere that is saturated intermittently or continuously. However, correctly speaking the term **delayed** ettringite formation only means that ettringite forms **later** and therefore it should include both *ESA* and *ISA* as shown in Table 3. The following terms are used in the present paper: "*ESA*" or "*ISA*" to indicate *DEF* related to *external* or *internal* sulfate attack, respectively.

EXTERNAL SULFATE ATTACK

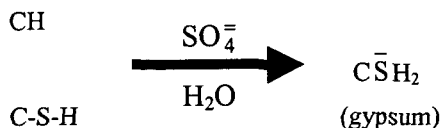
The *ESA*-induced damage, which is the traditional sulfate attack, is determined by the chemical interaction of a sulfate-rich soil or water with the cement paste. Soils containing sodium, potassium, magnesium, and calcium sulfate are the main sources of sulfate ions in groundwater. For *ESA* to occur the following three conditions must be fulfilled:

- **High permeability of concrete**
- **Sulfate-rich environment**
- **Presence of water**

A diagrammatic representation of the holistic approach (7) to the *ESA*-related damage can be illustrated through the help of Fig. 1 (8) where each contributory element corresponds to a circle. Each circle corresponds to only one of the three elements, and this situation does not present any risk at all for damage to a concrete structure. The area in the middle, where the three circles overlap, corresponds to situations of serious risk for the *ESA*-related damage since all the above-mentioned three elements are present. In the absence of one of these elements the *ESA*-related damage cannot occur. For instance, in porous and/or microcracked concrete not exposed to water, the *ESA*-related damage does not happen, even if sulfate ions are present in the environment because, in the absence of water, these ions — for instance in dry soil — cannot migrate through the interconnected pores of the concrete.

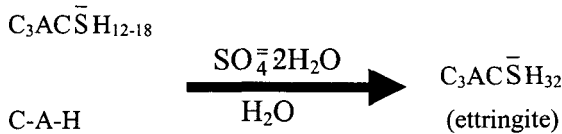
The *ESA*-related damage manifests itself in several forms including cracking, spalling, loss of strength and adhesion. The specific manifestation of the *ESA*-related damage depends on which one of the following three chemical processes is predominant:

- i) Sulfate attack on CH and C-S-H to form gypsum:



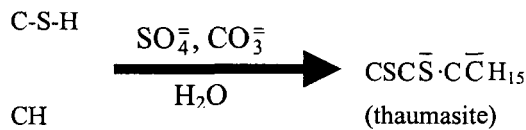
This process may cause expansion and spalling. However, its most important feature is the loss of strength and adhesion of the cement paste due to decalcification of C-S-H which is responsible for the binding capacity of the cement paste (2). This process may occur with all the sulfate salts (containing Na^+ , K^+ , Mg^{++}) except calcium sulfate.

- ii) Sulfate attack on calcium aluminate hydrates (*C-A-H*) and monosulfate hydrate ($C_3AC\bar{S}H_{12-18}$) to form ettringite:



This process is mainly responsible for cracking and spalling as a result of expansion produced by delayed ettringite formation. This process may occur with all the sulfate salts including calcium sulfate which acts directly on *C-A-H* and/or monosulfate hydrate.

- iii) Sulfate attack on *C-S-H* and *CH* in the presence of carbonate ions to form thaumasite:



The thaumasite formation is accompanied by the most severe loss of strength and adhesion, which is able to transform hardened concrete into a pulpy mass, since a significant part of *C-S-H* can be destroyed according to the reaction shown above. This process (9) may occur with every type of sulfate salts and is favored by humid atmospheres and low temperature (<10°C).

INTERNAL SULFATE ATTACK

The *ISA*-related damage is relatively "new" with respect to the "traditional" sulfate attack (*ESA*) since it was detected in the middle of 1980's in prestressed concrete railway ties (10). Two different mechanisms of *DEF* caused by *ISA* will be examined in this paper:

- Thermal decomposition and re-formation of ettringite in a saturated atmosphere at room temperature;
- Holistic approach based on microcracking, late sulfate release, and exposure to water.

Thermal Decomposition Mechanism

According to one school of thought (3,4,6) "normal" ettringite, formed at early age is destroyed by high-temperatures ($> 70^{\circ}\text{C}$) steam curing. Therefore, ettringite develops again at later ages in concrete structures exposed to water (either intermittently or permanently) and causes expansion or cracking. According to this school of thought (3,4) alkali silica reaction (*ASR*) has a particularly important effect in promoting expansion from *DEF* related to internal sulfate attack. In the presence of reactive aggregates, *ASR*-related expansion would begin during the heat treatment and cracking would immediately occur. Then ettringite forms in these cracks or other pre-existing weakness and is considered responsible for the subsequent additional expansion and cracking, if any. The following questions cannot find convincing answers from the above hypothesis (3,4):

- Why are prestressed concrete railway ties much more vulnerable to *ISA*-induced distress than other precast steam-treated concrete structures?
- Why before the 1980's *ISA*-related deterioration did not occur in concrete structures, particularly in prestressed concrete ties (10,11), even if steam curing at temperatures as high as 70°C was adopted at that time?
- Why is *ISA*-related distress so erratic (11) in the sense that, with the steam curing treatment, sometimes the damage occurs and sometimes it does not?
- Why *ISA*-induced distress does occur even in cast-in-place concrete structures cured at ordinary temperatures (10-14), although it is fair to admit that a higher frequency of this type of damage occurs in precast steam-cured concretes?

Before presenting an alternative hypothesis (11) which can give reasonable answers to the above questions, it is proper to give further comment in regard to the last question. According to Taylor (3) and Lawrence (4) *DEF* related to *ISA* may also occur in large sections and massive elements of non-steam-treated concrete, where the heat of hydration of the cement or an external heat source during service, may have caused 70°C or more temperature rise in concrete at early age. Accordingly *ISA*-related *DEF* could happen even in non-steamed-cured concrete provided that thermal decomposition of ettringite at or above 70°C had occurred.

According to the personal experience of the author as well as of other researchers (10-14), *ISA*-related *DEF* does occur even in non-steam-cured concrete at temperatures much lower than 70°C that are insufficient to cause thermal decomposition of the "normal" ettringite. This happens, for instance, in non-steam-cured "Friday concrete" ties which are not mass concrete structures. These concrete ties are manufactured on Friday and cured at room temperature because by Monday they would attain adequate

strength to allow for cutting the prestressing strands. Mielenz et al. (10) and Collepardi (11) found that *ISA*-related *DEF* may occur whether concrete ties are steam-cured or not, and this does not conform to the hypothesis (3,4) that *ISA*-related *DEF* is necessarily caused by thermal decomposition of ettringite at high temperatures ($>70^{\circ}\text{C}$).

Temperature measurements in concrete elements cured at room temperature, with different cement content, thickness and shape would determine whether or not thermal decomposition of ettringite can occur.

Holistic Approach Mechanism

A hypothesis exposed by the author (11), which satisfactorily addresses the above four questions, is based on the role played by the **late sulfate release** (from cement or aggregate) on delayed deposition of ettringite in the **pre-existing microcracks**. Intermittent or continuous **exposure to environmental water or saturated air** causes swelling or crystal growth of ettringite leading to crack opening, and subsequent damage in form of macrocracking.

The holistic approach according to this mechanism for the *ISA*-related *DEF* (11) is based on the presence of three essential elements:

- **Microcracking**
- **Late sulfate release**
- **Exposure to water**

This model can be represented in Fig. 2 where each element corresponds to a circle. The area in the middle, where the three circles overlap, corresponds to situations of serious risk of *ISA*-related damage since all the three needed elements of the system are present. In the absence of one of these elements *ISA*-related damage cannot occur.

The Role of Microcracking in *ISA*

Each of the three needed elements of the system can be related to numerous causes. For instance, concrete microcracking can be promoted by one or more of the following causes:

- Curing at high temperatures, excessive heating/cooling rate or too short preliminary curing at room temperature

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- *ASR* with microcracks around aggregate particles
- Weathering effects cycles including wetting/drying and heating/cooling changes
- Dynamic loads in service
- Plastic shrinkage in poorly cured slab structures
- Freezing/thawing cycles
- Excessively high porosity in aggregate particles
- Transition zone at the aggregate-cement or steel-cement interface
- Localized high stress in prestressed structures.

On the basis of this model, factors related to steam-curing (precuring time, heating and cooling rates, maximum temperature) play a subordinate role since they can only promote the occurrence of microcracking. This means that *ISA*-induced *DEF* may occur even in the absence of heat-curing provided that other causes, such as *ASR*, excessive stress, weathering effects, etc., can promote microcracks. On the other hand, everything being the same, steam curing at high temperatures causes additional risk of microcracking with respect to room temperature curing and therefore there is a higher frequency of occurrence of *ISA*-induced *DEF* in steam-treated concrete structures. This can also explain why in some laboratory studies (6, 15, 16) specimens cured at room temperature (in the absence of any mechanism responsible for microcracking) were not subjected to *ISA*-induced *DEF* in contrast to what occurred in the corresponding overheated steam-cured specimens.

According to the holistic approach, *ASR* is just one of the possible causes of microcracking promoting the ettringite deposition into pre-existing microcracks. *DEF* related to *ISA* was detected even in a paste cement system where microcracks could not be caused by *ASR* due to the absence of any reactive form of aggregate (11).

On the basis of this model, one can explain why prestressed concrete railway ties are more vulnerable to *DEF*-related damage induced by *ISA* than any other precast or cast-in-situ concrete structures. The higher frequency of *ISA*-related *DEF* occurrence in concrete ties is related to the excessive microcracking caused by localized stress induced by the cutting of the prestressing strands in concrete ties manufactured according to the "*long line method*" (11). On the other hand, when lower and more uniform stress distribution is adopted - such as in the "*anchored steel plate method*" - the frequency occurrence of the *ISA*-related *DEF* is much lower in the prestressed concrete ties produced in this manner (11). The evidence of this behavior as a function of the manufacturing process is a special feature of this model based on the holistic approach related to experiential knowledge rather than laboratory studies alone.

The Role of Late Sulfate Release in ISA

Modern cements, manufactured in kilns that burn sulfur-rich fuels or organic residues - such as old rubber tires - can incorporate large amounts of sulfate (up to about 2.5%) in the clinker phase (17). Some of this sulfur, according to Hime (14), is present as relatively slowly soluble sulfate. It is not available, for the early ettringite formation according to the reaction [1], but would be available **later** to produce delayed ettringite formation.

According to the holistic model (11), delayed ettringite formation, related to *late sulfate release*, can occur only if pre-existing voids, primarily in form of microcracks, are available. This important relationship between *late sulfate release* and pre-existing microcracks in determining the *ISA*-related *DEF*, again is based on the experiential knowledge related to the manufacturing process of prestressed railway ties. For instance, in many concrete ties, where there were complaints for the *ISA*-related *DEF*, microcracks were detected, by using an optical microscope, either immediately after cutting the prestressing streams or later on in stock-piled products. Then microcracks transformed into macrocracks (visible to the naked eye) and delayed deposition of ettringite into these cracks was detected by XRD and SEM. In addition to the slowly soluble sulfate in the clinker phase, related to the high-sulfur fuels or organic residues, *late sulfate release* can be attribute to other possible causes:

- Sulfate release from gypsum-contaminated aggregates, for the relatively coarse size of the calcium sulfate particles, is not immediately available for *EEF* and then can feed *DEF*.
- Thermal decomposition (3,4,6,15,18) of "normal" ettringite in overheated concrete is potentially another internal source of late sulfate release.
- Sulfate ions are slowly released from that adsorbed on the *C-S-H* phase in high temperature steam-cured concretes (19).

The last two potential sources of late sulfate release were theoretically available even before the 1970's, but then they cannot explain why there was a significant increase in the incidence of *ISA*-related *DEF* during the last two decades, particularly in the manufacture of prestressed concrete ties. However, they are included in the holistic model of the present paper (Fig. 2) as other potential sulfate resources in addition to those considered in a previous paper (11).

The *late sulfate release*, related to the clinker sulfate increase with the use of high-sulfur fuels or organic residues in the last two decades, can more easily justify why there was an increase in *ISA*-induced distress incidence, from the 1970s to the 1980s, particularly in prestressed concrete ties more prone to microcracking from the manufacturing process itself. On the other hand, *ISA*

induced damage seems to be an exceptional and discontinuous phenomenon rather than a general and continuous occurrence. This erratic occurrence of the *ISA*-related damage can be related to the intermittent use of sulfur-rich organic residues in the clinker kiln or to the change in the sulfur content of ordinary fuels. In many present-day clinker kilns there are poly-functional burning systems which are capable of using either gaseous or liquid hydrocarbons, as well as solid small-particle coals, depending on the cheapest source of available fuels. The sulfur content of these different fuels can change and cause unwitting variations of sulfate incorporated in the clinker phase.

In agreement with the school of thought (3, 4) based on thermal decomposition of "normal" ettringite ($>70^{\circ}\text{C}$) as responsible for *ISA*-related *DEF*, some researchers (17, 20-26) think that the sulfate content of the clinker phase can be ruled out as a source for the delayed ettringite formation at ambient temperature. According to these researchers, even for clinkers with high sulfur levels ($\text{SO}_3 = 2.5\text{-}3\%$) all the clinker sulfate would be rapidly available to form "normal" ettringite within several minutes (25) or few days at most (26).

There is another reason why the important role played by *late sulfate release* from the clinker phase is denied by the researchers in favor of the thermal decomposition of "normal" ettringite ($> 70^{\circ}\text{C}$) as responsible for *ISA*-related *DEF* (3, 4, 17, 20-24): according to this school of thought laboratory studies indicate that even with sulfur-rich clinkers there is no risk of *ISA*-related *DEF* at ambient temperature. However, according to the holistic approach presented by Fig. 2, *late sulfate release* by itself cannot cause any *ISA* in the absence of water exposure and/or pre-existing microcracking. In laboratory studies only concrete specimens subjected to steam-curing (particularly those cured at high temperatures, with excessive heating/cooling rate or too short preliminary curing at room temperature) can microcrack. Other causes of microcracking at room temperature (restrained hygro-thermal changes, excessive and/or non-uniform stress in prestressed structures, loading in service, etc.) might be present in the field concrete structures. Therefore, it should not be surprising that *ISA*-related *DEF* can occur *even* in concrete structures cured at ambient temperature in the field (10-14), whereas it can occur *only* in overheated concrete specimens when studied in laboratory (17, 20-24).

The Role of Exposure to Water in *ISA*

The third needed element for the *ISA*-induced damage is the intermittent or continuous exposure to water or humid air (Fig.2). The presence of water filling the pore system of concrete is essential for the migration of reactant ions — $\text{SO}_4^{=2}$, $\text{Al}(\text{OH})_4^-$ and Ca^{+2} — up to microcracks where ettringite deposition can occur. Field experience indicates that concrete ties exposed to the rain and sun alternate actions (on the sides and especially at the tops of outdoor storage

stacks) are more severely damaged by *ISA* than those exposed to rain but in a permanent shadow condition (11, 27). This different behavior could be attributed to the important role played by the degree of supersaturation in favoring the deposition of ettringite. On the other hand there is experiential knowledge (10, 28) that microcracked concrete ties, with the same cement source and concrete composition causing *ISA*-induced damage in other wet concrete structures, do not show such type of deterioration when they are protected from contact with water (concrete products protected by hydrophobic treatments or stock-piled ties below the outdoor storage stacks). This confirms the important role played by water in the migration of reactant ions from the cement paste up to the site of microcracks, and then resulting in the deposition of ettringite crystals into the microcracks and the subsequent crack opening.

CONCLUDING REMARKS AND RECOMMENDATIONS

Ettringite formation by reaction of internal or external sulfate with anhydrous or hydrated calcium aluminates has an expansive character. However, not necessarily the ettringite formation produces damaging expansion. When it occurs *immediately* (within several hours) in a fresh mixture (*early ettringite formation EEF*), there is no expansion. *EEF* is advantageously used for setting time retardation of portland cement.

On the other hand, when ettringite forms *heterogeneously at late ages* (after months or years), — *delayed ettringite formation (DEF)* — in a rigid hardened concrete, it can produce cracking and spalling.

There are two different types of *DEF*-related damages depending on the sulfate source: external or internal sulfate attack. *External sulfate attack (ESA)* usually occurs when *environmental sulfate* (from water or soil) penetrates into a concrete structure. It can be prevented or reduced by using impermeable concrete.

Internal sulfate attack (ISA) occurs in a sulfate free-environment by the *late sulfate ions release* from either cement or gypsum-contaminated aggregates. Of these two internal sulfate sources, the latter is relatively rare because it can be prevented by controlling the sulfate content of the aggregates. The *ISA*-induced damaged caused by the late sulfate release from the cement can be related either to thermal decomposition of ettringite and sulfate release from *C-S-H* or to the increase in the sulfate content of the clinker phase, particularly during the last two decades, due to the use of sulfur-rich fuels or organic residues that are being burned in clinker kilns to destroy environmentally harmful products in a safe and cost-effective way. Unfortunately, it is not easy to detect the sulfate content of the clinker phase since only the total sulfate content — including that from gypsum acting as set regulator — can be easily and quickly determined in a routine control.

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The hypothesis of thermal decomposition of ettringite in steam-cured concrete, as the main source of *DEF* caused by *ISA*, is less convincing for at least two reasons:

- *DEF* caused by *ISA* occurs even in non-steamed concrete structures at room temperature
- *DEF* caused by *ISA* was not detected before the 1980s, when millions of prestressed concrete ties were manufactured without any significant damage in service, although steam-curing was also adopted for these concrete products.

Hopefully, to confirm or reject the thermal decomposition mechanism, future research should address the two above aspects:

- i) Laboratory and field tests to measure the maximum temperature attainable in concrete structure as a function of content and type of cement, thickness and shape of concrete elements, environmental temperature, etc.
- ii) Statistical studies in the field of precast concrete industries, particularly in the area of prestressed concrete ties, to investigate whether other factors dealing with the process (maximum temperature, heating/cooling rates, preliminary curing, etc.) were modified from 1970's to 1980's, so that they could change the incidence of the *ISA*-related distress in that period of time.

Independently of the mechanism, the risk of *ISA* induced damage can be prevented provided that one of the following essential elements is precluded:

- **Microcracking**
- **Late sulfate release**
- **Exposure to water**

Apparently the easiest preventive method would relate to water exposure. However, in practice, permanent protection of concrete structures from exposure to water is very expensive by using the present day available hydrophobic or impermeable coatings.

Microcracking can be significantly reduced to harmless level by adopting adequate measurements during the design and the execution phases. For instance, precast concrete ties with lower and more homogeneous stress distribution, deriving from the prestressing process itself, should be taken into careful consideration by design engineers. On the other hand, producers of precast concrete elements should take into account that steam-curing with lower thermal gradients, from controlled heating and cooling rates, can also reduce microcracking and consequently the *ISA*-induced damage risk.

Finally, cement producers should investigate why certain cements are more prone to *ISA*-damage than others. In particular, they should study the role played by the total sulfate content of the cement as well as that of the clinker

phase in sound and microcracked concrete elements, cured at room or high temperature, and then exposed to water or saturated atmosphere. In the meantime, blended cements — particularly pozzolanic and blast furnace slag cements — should be preferred (15, 29) instead of high early strength portland cements, in the manufacture of concrete products, such as prestressed concrete ties, which are more vulnerable to delayed ettringite formation.

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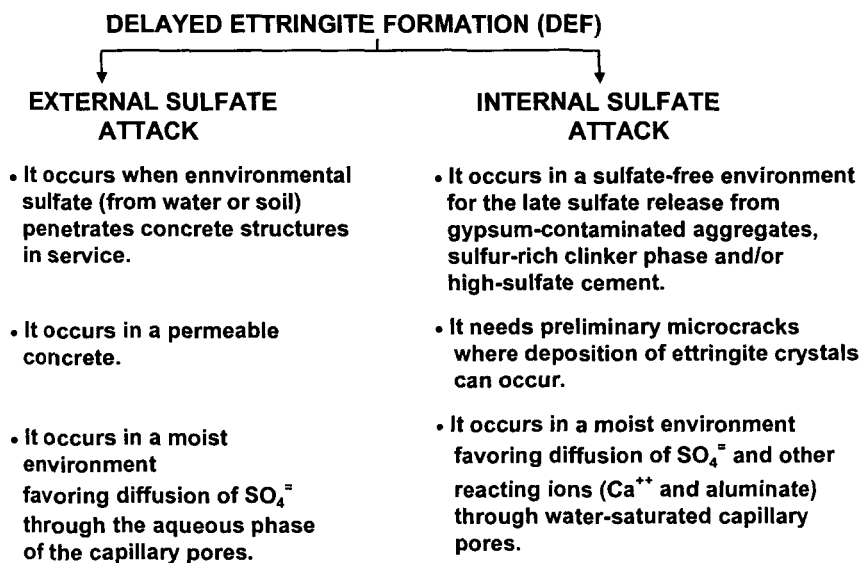
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Table 1 - Early Ettringite Formation (EEF) "Normal" Ettringite.

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| <ul style="list-style-type: none"> • It occurs <u>homogeneously</u> and <u>immediately</u> (within hrs or days). |
| <ul style="list-style-type: none"> • The related expansion, if any, does not cause a localized and disruptive action since it occurs in a deformable concrete. |
| <ul style="list-style-type: none"> • It is due to ground gypsum which reacts with calcium aluminates of the portland clinker phase (set regulation) or to sulfate-based expansive agents in shrinkage compensating concretes. |

Table 2 - Delayed Ettringite Formation (DEF) "Secondary" Ettringite.

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|--|
| <ul style="list-style-type: none"> • It occurs <u>heterogeneously</u> and <u>later</u> (after months or years). |
| <ul style="list-style-type: none"> • The related expansion produces cracking, spalling, and strength loss since it occurs in a rigid, stiff, hardened concrete. |
| <ul style="list-style-type: none"> • Its damaging effect is related to environmental or internal sulfate source (sulfate attack). |

Table 3 - Delayed ettringite formation by external and internal sulfate attack.

DEF RELATED TO EXTERNAL SULFATE ATTACK (ESA)

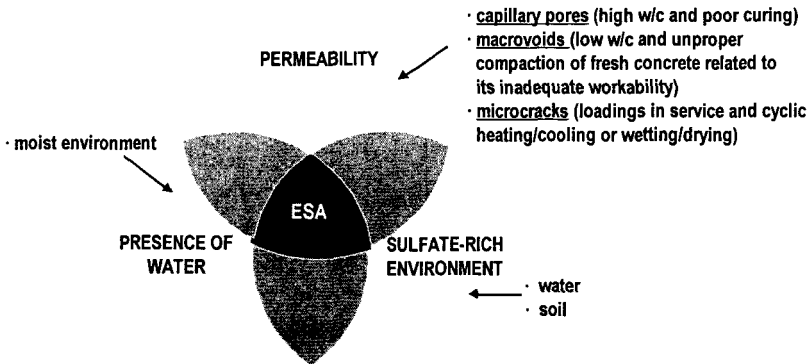


Fig. 1 – Ternary representation of the DEF related to ESA.

DEF RELATED TO INTERNAL SULFATE ATTACK (ISA)

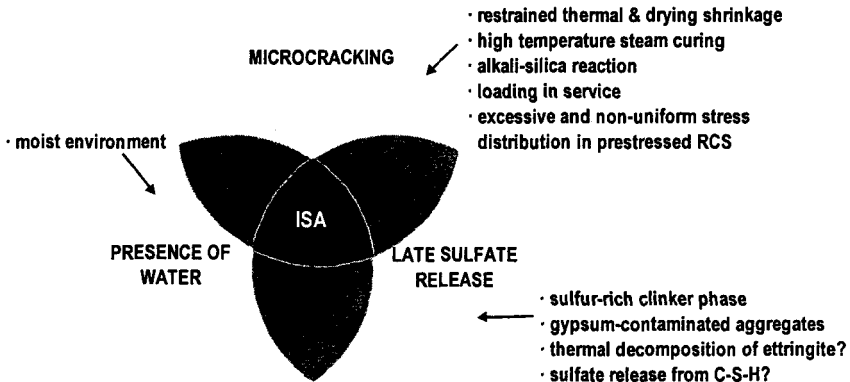


Fig. 2 – Ternary representation of the DEF related to ISA.

Counteracting Retardation of Cement Setting By Other Admixtures with Calcium Nitrate

by H. Justnes and B. G. Petersen

Synopsis: High dosages of lignosulphonates for super-plasticizing are prohibited by the simultaneous setting retardation. However, the set retardation of lignosulphonates can be counteracted by calcium nitrate without destroying the rheology. Thus, such combinations can be cost-effective alternatives to super-plasticizers. The effect of different calcium nitrate dosages on 5 different lignosulphonates is documented by Fann viscosity, flow resistance and setting time on two different cement pastes.

It has also been shown that calcium nitrate is capable of counteracting the effect of strong setting retarders like citric acid etc. Another application can thus be over-retardation of concrete for long transport from the concrete ready mix plant, followed by activation by adding calcium nitrate to the revolving drum of the concrete truck when arriving at, or being close to, the construction site.

Keywords: admixtures; calcium nitrates; retardation

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INTRODUCTION

The development in super-plasticizers goes towards more synthetic and complicated materials that, although more effective, are quite expensive and are based on refined chemicals. The plasticizer lignosulphonate, on the other hand, is a by-product from the pulping industry and is derived from natural wood chips as a raw material. However, lignosulphonate cannot be used in high enough dosages to act as a super-plasticizer due to the simultaneous retarding effect on cement setting. For this reason, typical high performance (i.e. low W/C) concrete technology in Norway is in practice based on a cheap lignosulphonate topped-up by a more expensive super-plasticizer to obtain an economic solution to desired workability.

The target of the present study was to investigate if it was possible to obtain sufficient workability with a high dosage of lignosulphonate and counteract the retarding effect by adding the relatively cheap setting accelerator calcium nitrate (1). A second objective was to see if calcium nitrate could be used to counteract retardation of rather strong chemical retarders. Such a combination could then be used in the transport of retarded ready mixed concrete in long or unpredictable hauls and could be activated close to the construction site by adding calcium nitrate in the revolving drum of the truck.

EXPERIMENTAL

Materials

A high strength portland cement (HSC) was used with composition 64.01 % CaO (C), 21.47 % SiO₂ (S), 4.17 % Al₂O₃ (A), 3.40 % Fe₂O₃ (F), 2.77 % SO₃ (S̄), 1.74 % MgO, 0.48 % K₂O and 0.29 % Na₂O. This corresponds to the