

Accepted Manuscript

Title: Freeze/thaw protection of concrete with optimum rubber crumb content.

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PII: S0959-6526(11)00384-2

DOI: [10.1016/j.jclepro.2011.10.013](https://doi.org/10.1016/j.jclepro.2011.10.013)

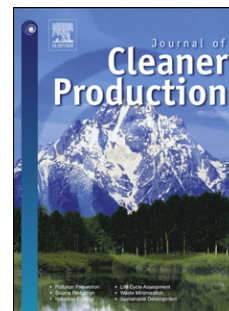
Reference: JCLP 2599

To appear in: *Journal of Cleaner Production*

Received Date: 8 July 2011

Revised Date: 10 October 2011

Accepted Date: 10 October 2011



Please cite this article as: Richardson AE, Coventry KA, Ward G. Freeze/thaw protection of concrete with optimum rubber crumb content., *Journal of Cleaner Production* (2011), doi: 10.1016/j.jclepro.2011.10.013

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Freeze/thaw protection of concrete with optimum rubber crumb content.

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Abstract

This research looks at utilising an optimum quantity of rubber crumb as an air entraining admixture in concrete, thus providing maximum freeze thaw protection and maximum strength. Microscopic and chemical analysis was carried out on the rubber sample to investigate how rubber crumb entrains air and reacts with the surrounding concrete. The work contained two pilot studies that informed the main test methodology. The pilot studies examined the air content/compressive strength relationship (1) and freeze/thaw cycle durations (2). Pilot study 1 informed the main test program by identifying an optimum addition of rubber crumb to a concrete mix, which was found to be 0.6% by weight of concrete. The main test investigated the use of rubber crumb in providing freeze-thaw protection of a C40 concrete mix after 3 days of curing.

A freeze-thaw test was carried out on three separate batches of concrete containing washed rubber crumb, unwashed rubber crumb and plain concrete respectively. It was found rubber crumb was effective in providing freeze/thaw protection in both cases.

This work builds on recent work to identify the best practical solution for reducing waste and providing the maximum freeze/thaw protection for a cleaner production process.

Key Words

Waste reduction, rubber crumb, freeze-thaw protection, durability, optimum rubber addition, cleaner production

1.0 Introduction

According to Dovi, *et al.* (2009), “Current and future developments in National and World economies are closely connected to sustainable efficient and safe usage of raw materials and upon energy based on cleaner production concepts and approaches that are ecologically and economically appropriate for the short and for the long term future of society”. Worldwide generation of waste tyres amounts to 5 million tones per year, representing 2% of total annual solid waste (Singh *et al.* 2009).

The UK Government is driving a sustainability culture (WRAP 2011) and any additive that creates a more durable concrete product of enhanced environmental credentials is worthy of investigation. Long, *et al.* (2001:65) state that, “It has been estimated that the value of the infrastructure and built environment represents 50% of the national wealth within most European countries, because of the degree and rate of degradation of the built environment in Europe, it is of enormous economic and technical importance to provide a low maintenance environment”. This view is also taken by Mulheron (2001:1) who states that, “The need to improve our ability to both understand the mechanisms by which deterioration occurs, and the impact that methods of preventing deterioration have on subsequent material performance, is driven by the high cost of maintaining an ageing infrastructure”.

If rubber crumb is found to be effective in providing enhanced durability and is adopted as a concrete additive, the reduction in maintenance and remedial work to concrete may be significant. The size of the UK concrete repair sector is estimated to exceed 3% of the entire construction industry output (Waterman 2006), which currently stands at £106 million GBP (Office of National Statistics, 2011). In addition, “with the world pouring around 5 billion tonnes of concrete a year – nearly one tonne per person per year – concrete is probably the most common material in modern construction” (Kernan, 2003). If this concrete can have low life cycle costs due to enhanced durability, this will have a lower environmental impact upon our world, with subsequent benefits of reduced carbon emissions and careful use of natural finite resources.

“The UK produces 487,000 tonnes of used-tyres each year that have to be reused or disposed of...”(Environment Agency, 2010). The Landfill Directive has banned the land filling of whole used tyres since 2003 and shredded tyres since 2006 (Defra, 2010). At present, end of life tyres have varied uses such as; carpet underlay, and as tyre derived fuel (Singh *et al.* 2009) used in cement kilns (WRAP 2006 and Strazza *et al.* 2011) or in asphalt (Milanez and

Bühns 2009) but this does not fully utilise the volume of tyre waste being produced. The economic benefits of utilising rubber crumb to provide a durable concrete provide a significant long term benefit to society, due to lower life cycle costs. Adhikara, *et al.* (2000) suggest that, “Among various methods of disposal of scrap/waste rubber products, recycling or reclaiming of rubber is the most positive approach, because it not only saves our limited resource fossil feedstock but also maintains our environmental quality”. However the use of rubber crumb as an additive to coal causes air pollution that arises from the combustion process (Fang *et al.* 2001). Rubber crumb contains volatile components that need to be re-burnt to meet the requirements of the Large Combustion Plant Directive (LCPD 2001/80/EC) (Singh *et al.* 2009) and this requires a complex infrastructure to deal with the problems of corrosive elements and particulate control. Addressing the potential use of rubber crumb as a concrete additive, avoids the environmental concerns attributed to its disposal. Thus this paper addresses the potential of rubber crumb as an air entrainment agent.

Introducing an air entrainment agent into concrete is known to reduce the compressive strength. For every 1% of additional air entrained, the concrete strength will fall by typically 5 to 6% (Cement Admixtures Association, 2006) and this effect is also exhibited on the introduction of rubber crumb. Ganjian *et al* (2008:1832) found that, on adding 5% by volume of powder rubber as a sand replacement, the compressive strength was reduced by approximately 5%. Savas *et al.* (1996), Benazzouk and Queneudec, (2002) Paine and Dhir (2010) all carried out work on rubber crumb in concrete with regard to the freeze thaw resistance of concrete. Each researcher showed a noticeable increase in the durability factor of the concrete samples containing rubber but no research has considered the optimum quantity of rubber crumb required to maximise the compressive strength and freeze/thaw protection of the concrete produced.

Freeze-thaw damage can occur in concrete at any stage, between pouring and achieving full cure. To provide freeze-thaw protection, current practices use air entraining admixtures which induce pockets of air into the concrete. These air pockets act as expansion chambers during the expansion and contraction of the concrete when subject to freeze-thaw cycles. This study aims to discover the possibility of achieving an optimum rubberised concrete mix that provides maximum freeze-thaw protection, whilst minimising the compressive strength loss.

2.0 Methodology

A variety of current freeze/thaw standards were adapted to carry out this research. The British Standard CEN/TR 15177:2006 defined the duration of the freeze-thaw cycles, while the

American Society for Testing and Materials (ASTM C 666) informed the dry freeze and wet thaw procedure as defined within ASTM 666 as Procedure B. The calculations of the durability factor is common to both standards, in that it considers the percentage change from the original value to the final value.

The main freeze/thaw and pilot test was carried out using six 100 mm cubes per concrete type and the control samples used six cubes for the initial strength and six cubes for the final strength at the end of the freeze/thaw program. Curing was carried out for three days in the curing tank at 20°C prior to starting the freeze/thaw test.

The material specification and manufacturing process for the cubes tested are considered below. The methodology pursued, considered the determination of the following parameters: concrete density (Concrete Society, TR 32: 1989), the characteristic compressive strength (BS EN 12390-3:2002), pulse velocity as a relative dynamic modulus (BS 1881: Part 203: 1986), mass lost due to freeze/thaw action and the durability factors of the concrete test specimens (ASTM 666C:97). The durability factor was determined at the end of the freeze-thaw test and is shown in Equation 1.

$$DF = PN/M \quad (1)$$

DF = Durability factor of test specimen

P = Relative dynamic modulus of elasticity at N cycles %

N = Number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and

M = Specified number of cycles at which the exposure is to be terminated

P is determined using Equation 2.

$$P = (n_1^2/n^2) \times 100 \quad (2)$$

P = Relative dynamic modulus of elasticity after c cycles of freezing and thawing as a %

n = fundamental transverse frequency at 0 cycles of freezing and thawing

n₁ = fundamental transverse frequency at c cycles of freezing and thawing

(ASTM C 666, 1997:327, p.4)

2.1 Specification for the rubber crumb concrete

2.2 Mix Design

A normal C40 concrete mix was chosen as a structural concrete grade that is widely used throughout the industry. The C40 design mix used is shown in Table 1, was designed by ready mix concrete technical department Cemex (2010) UK to provide the required design strength.

Material	Quantity (kg/m ³)
Cement (CEM1- Ferrocrete)	403
Sand (0-4mm)	837
Aggregate (4-10mm)	336
Aggregate (10-20mm)	621
Water cement ratio 0.45%	181 (litres)
Rubber crumb	To be determined

Table 1 - C40 Mix Design (Cemex 2010)

Figure 1 illustrates the concrete batching and cube manufacture schedule. The percentage of rubber crumb addition to mixes A and B was informed by the Pilot Study presented in Section 3.

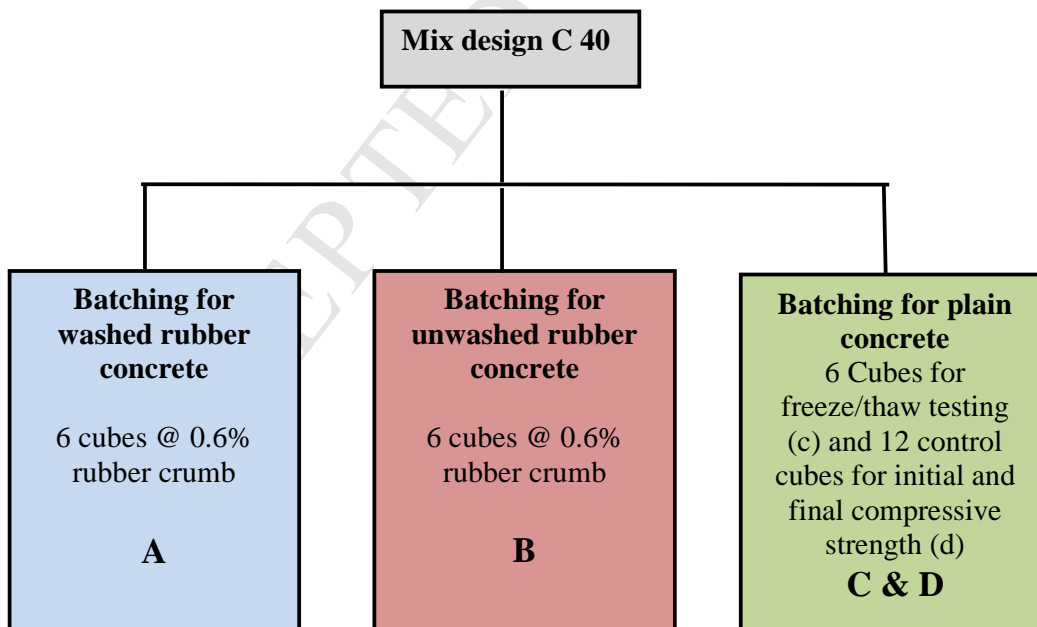


Figure 1 - Concrete Batching Programme

2.3 Aggregate and rubber crumb grading

The fine (4 – 10mm) and coarse (10 - 20mm) aggregate were gap graded. The rubber crumb displayed a tendency towards a single size particle as shown in Figure 2. The rubber analysis showed 100% of the rubber sample passed through both the 3.35 and 2.36mm sieves. The majority of the sample was retained in the smaller sieves ranging from 0.6 – 0.15mm therefore the rubber sample used in this study can be classified in part as rubber crumb, according to Siddique and Naik (2004).

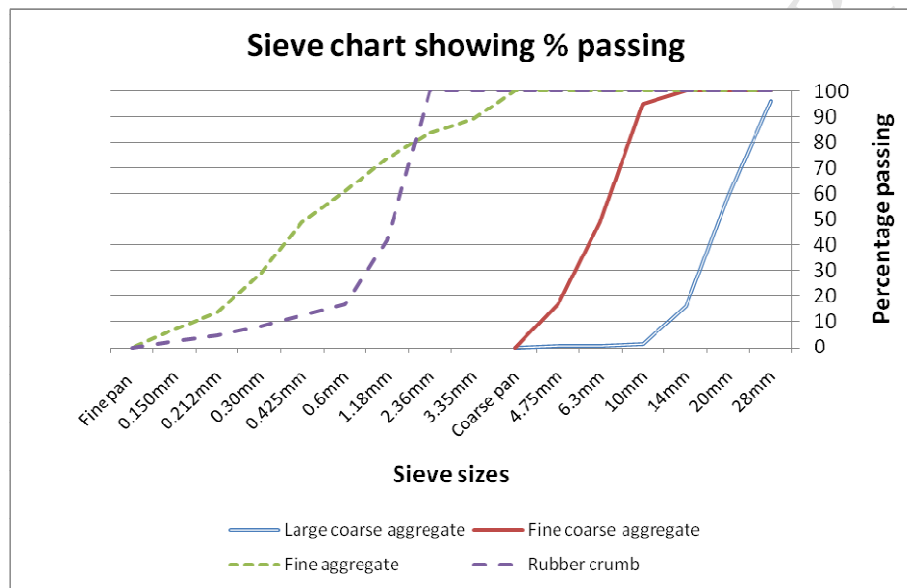


Figure 2 – Rubber crumb and aggregate sieve chart

2.4 Crumb specification

The rubber crumb content was determined from the Pilot Study 1. Rubber crumb was considered to be an additive to the design mix and not a sand replacement as adopted by Pelisser et al. (2011). Replacing sand with rubber crumb has the effect of reducing the compressive strength and elastic modulus. These are two material qualities that this study is trying to optimise when using rubber crumb in concrete through the determination of an optimum design mix. Due to the logistical constraints of freezer space and the health and safety considerations resulting from the manual handling of the cubes, the cube dimensions were constrained to 100mm. Adopting 100mm cubes, ensures that the distance between the exterior surface and the cube core is not large enough to limit the surface water absorption to the cube's core (Li et al. 2009). In addition to this, the surface area to volume ratio of a

100mm cube is 2.25 times greater than a 150mm cube, making the 100mm cubes more vulnerable and prone to damage than a 150mm cube.

2.4 Electron microscope examination of the rubber crumb

The Leica S6D scanning electron microscope (SEM) was used to examine the rubber crumb particles used within the study. To understand how the rubber crumb particles may combine with the cement paste, an examination was undertaken to evaluate the morphology of the rubber crumb particles. Figures 3 and 4 show the rubber sample at two magnifications and allows for the particle size and shape to be analysed, from which assumptions can be made with regard to surface tension and air entrainment due to the shape of the rubber crumb.

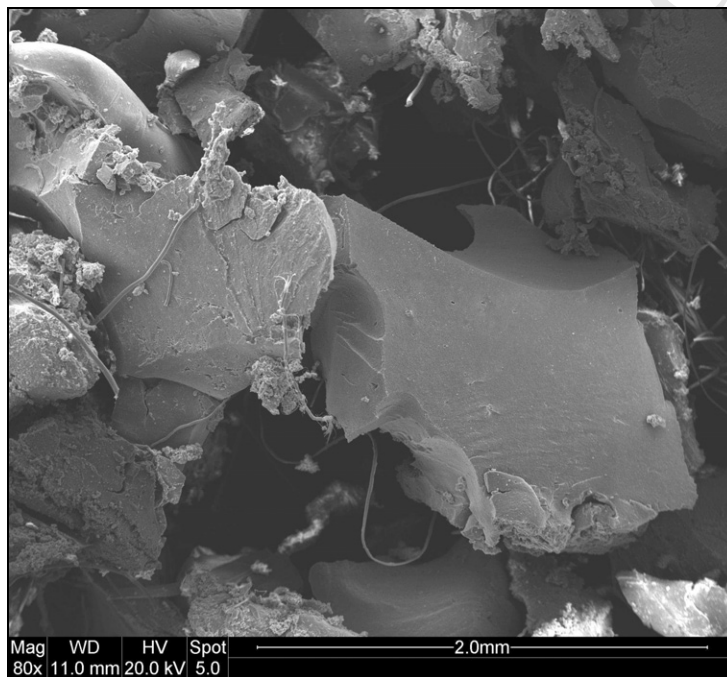


Figure 3 – Rubber crumb particle at 80x magnification

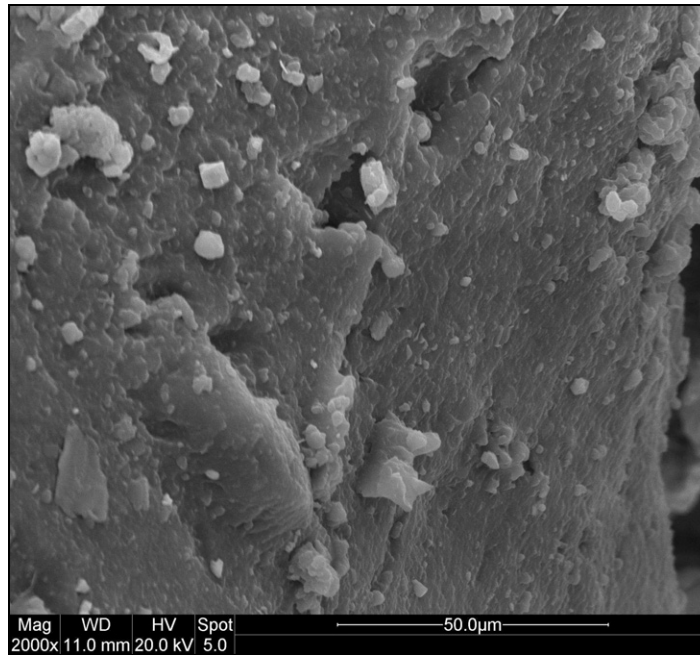


Figure 4 – Rubber crumb particle at 2000x magnification

The results of this rubber particle analysis indicate that, during the processing of the tyre from which rubber crumb is created, the rubber is broken down into small jagged shapes which have a rough surface. ~~It is believed~~ Benazzouk et al. (2008) suggest, that these irregular profile shapes entrap air during the batching process which could explain how the rubber crumb provides air entrainment when added to concrete. Pelisser et al. (2011), in their study of the effect of alkali activation on the compressive strength of concrete using silica fume and rubber particles over a range of sizes, discovered the occurrence of a gap at the interfacial zone between the rubber crumb and the concrete/cement paste. The occurrence of this zone in hardened concrete will further contribute to the provision of a pressure release system, enabling freeze/thaw protection.

2.5 Chemical Analysis

Figure 5 displays the results from the Energy Dispersive Spectroscopy (EDS) image analysis that was carried out on the rubber crumb sample. It displays all the elements that were present and the higher peaks indicate a greater content of individual elements. The high incidence of silicone in the rubber crumb, indicates the tyres used were from a developed country, as silicon is used to extend tyre life (Milanez and Bührs 2009).

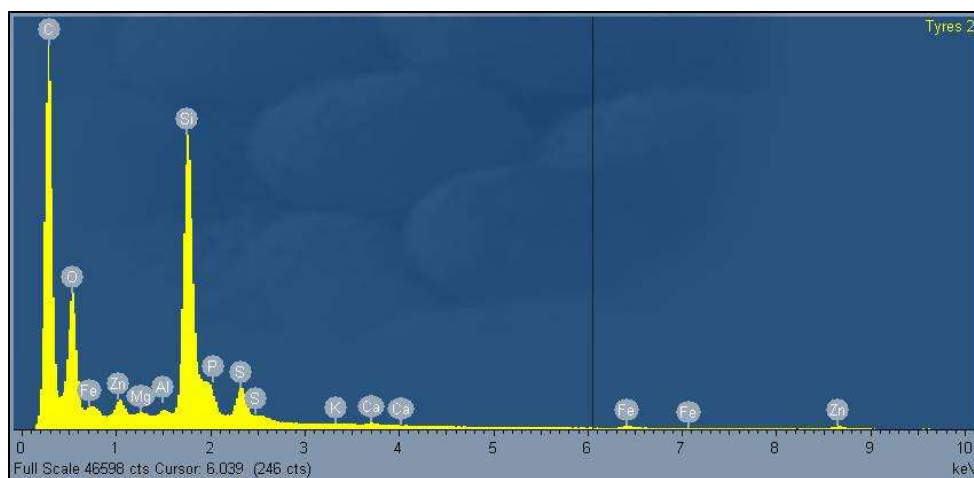


Figure 5 – Chemical Analysis Results of the Rubber Sample

The chemical breakdown as displayed using EDS in Figure 5 is representative of a common tyre compound; in that the approximate proportions mirror those as shown using EDS by Pelisser et al. (2009). This test indicates that there are no additional elements present that would adversely affect the research. The chemical analysis identified that there was high level of silicon (Si) present in the rubber sample and this would explain why the rubber and water did not mix during batching; as the water would be repelled by this hydrophobic, naturally water resistant, material. Table 2 presents the chemical composition of the rubber crumb which was determined using EDS.

Composition of elements		Percentage
Carbon	C	75.32
Oxygen	O	6.95
Magnesium	Mg	0.097
Aluminium	Al	0.085
Silicon	Si	22.17
Phosphorus	P	0.25
Sulfur	S	1.00
Potassium	K	0.015
Calcium	Ca	0.074
Iron	Fe	0.25
Cobalt	Co	0.002
Zinc	Zn	0.64
Total		99.98

Table 2 Chemical properties

Table 2 provides a complete overview of the chemical properties of the rubber crumb as used.

A full analysis of the rubber crumb can be found in The Journal of Green Building, (Richardson et al 2011) and the results are obtained from ASTM D 412, ASTM D – 2084, and IS 7490:1997. The work presented here expands upon earlier work by Richardson et al (2011) carried out on the same rubber crumb particles.

3.0 Pilot studies and material analysis

3.1 Optimum air content/compressive strength determination (Pilot study 1)

A pilot test was required to determine the optimum percentage of rubber crumb addition that displayed the highest compressive strength and highest percentage of air entrainment. The pilot study was informed by the work of; Ganjian et al (2009), Katib and Bayomy (1999), Topçu (1995) and Biel and Lee (1996) who all suggest that 5% rubber addition (by volume) provides noticeable air entrainment while concrete strength is not affected.

Six batches of six concrete cubes were produced, five of these were with a rubber crumb addition ranging from 0.3% - 1.5% by weight (solid density) produced in 0.3% increments. A single air content test (BS EN 12350-7:2009) per batch was carried out on the fresh concrete and was used to determine the percentage of air contained within the plastic concrete. In conjunction to the air test, a compressive strength was carried out at 28 days using BS EN: 12390 – 3, 2002. The results of these tests are shown in Table 3. The standard deviation values of the compressive strength were as follows: plain concrete (1.34), 0.3% rubber crumb concrete (1.42), 0.6% rubber crumb concrete (1.09), 0.9% rubber crumb concrete (0.96), 1.2% rubber crumb concrete (1.48) and 1.5% rubber crumb concrete (1.53). There was very little scatter in the results. The rubber crumb concrete mixes had density standard deviations ranging between 14.02 and 14.58, again with very little scatter.

% Rubber addition (by weight)	Air content (%)	Average compressive stress (N/mm ²)	Average density (kg/m ³)	Slump (mm)
0 (Plain)	2.0	64.9	2412	55
0.3	2.5	59.9	2402	55
0.6	2.5	63.5	2394	50
0.9	2.5	55.8	2378	50
1.2	2.7	56.2	2375	55
1.5	2.7	51.5	2367	70

Table 3 - Air/strength - Pilot Study Results

The overall reduction in density between plain concrete and rubber crumb concrete with 1.5% addition by weight was 1.9% and the air content differential was 0.7%. A progressive reduction in density was observed with each incremental addition of rubber crumb and a 0.5% increase in air content was recorded when comparing plain and 0.3% rubber crumb concrete. The design mix containing 0.6% rubber crumb by weight, provided the highest compressive strength of all the rubberised mixes. This result informed the rationale for the selection of the design mix adopted in the primary investigation.

3.2 Determine optimum freeze/thaw operational procedures (Pilot study 2)

Pilot study 2 used a thermometer embedded in the centre of a concrete test cube with a silicon seal. Using ASTM C666 - 97; to inform optimum freezing and thawing durations, a thermometer was embedded in the centre of a concrete test cube with a silicon seal to determine the cycle times and obtain temperature values between -17.8 to 4.4°C . The results from this pilot study were then used to create a testing schedule that entailed a 7 hour freezing time and a 20 minute water thawing period to comply with the ASTM requirement.

4.0 Freeze/thaw test results

4.1 Batching concrete

A slump test was used to determine consistency with batches A and B achieving a 50mm true slump and batches C and D a 70mm true slump. The results of this test indicate that the rubberised concrete achieved a reduced slump and the results are in keeping with the findings of Eldin and Senouci (1993), and Kathib and Bayomy (1999).

4.2 Compressive strength and density

The density of the batched concrete was determined and recorded as follows: washed rubber crumb concrete (A) 2129.6 kg/m^3 , unwashed rubber crumb (B) 2162.8 kg/m^3 , plain (C) 2338.3 kg/m^3 and plain control (D) 2326.1 kg/m^3 and the respective standard deviations were 14.55, 14.02, 51.41, and 12.18. The lower density of the rubber crumb concrete can be attributed to the entrained air. The relative weight of concrete (TR 32 – 1989) can be used to determine the air content, when air entrained concrete can be compared to a plain reference sample. The rubber crumb concrete displayed a lower weight (-186 kg), 8% less than the

plain concrete control samples and this is an indication of air content and lower density due to the light weight rubber crumb particles.

After 3 days of curing, the compressive strength was 11.1 N/mm² (Batch D - Initial) at which point the freeze/thaw cycles began. After 56 freeze/thaw cycles the compressive strength was 10.8 N/mm² for washed rubber crumb concrete (Batch A), 9.3 N/mm² for unwashed rubber crumb concrete (Batch B) and 3.6 N/mm² for plain concrete (Batch C). The standard deviations for the respective results are 0.25, 0.41 and 0.6. The control mix achieved a compressive strength at the end of the freeze/thaw testing period of 43 N/mm² (Batch D – Final) with a standard deviation of 1.09. Figure 6 compares the initial and final strengths of batch D and the final freeze thaw strength of batches A, B and C. ~~all concrete batches.~~

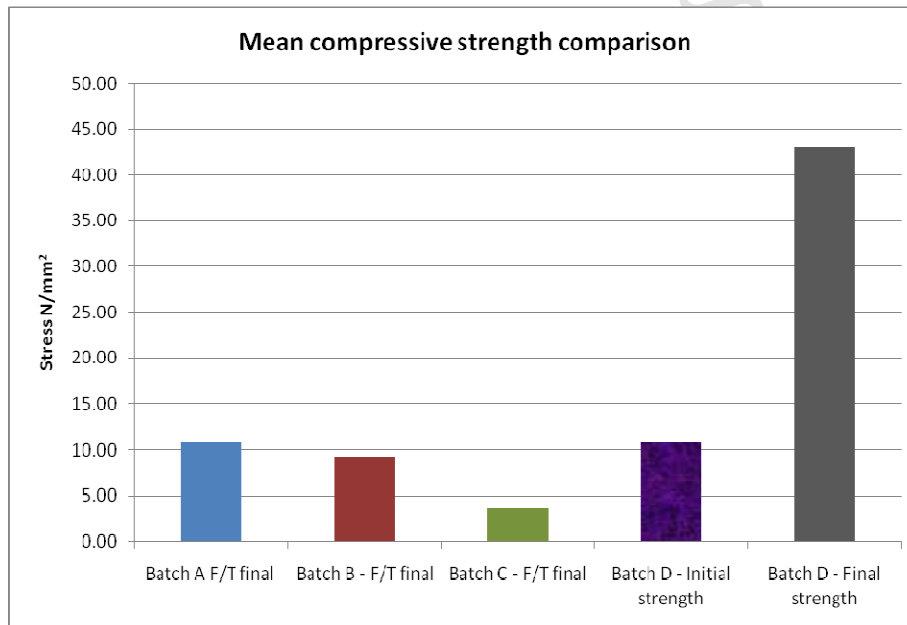


Figure 6 – Average compressive strength comparison

When comparing the final compressive strengths, it is evident that the rubber concrete specimens subjected to the freeze-thaw cycles, suffered minor strength loss when compared to the initial compressive strength. The plain concrete was prone to damage whereas the concrete with rubber crumb survived very well. Batch A and B showed similar strength loss. The small additional strength seen in batch A could be attributed to the washing of the rubber crumb prior to batching. These results would support the theory, shared by many researchers such as Eldin and Senouci (1993) that washing the rubber crumb with water helps the bonding of the rubber and cement. This enhanced bond results in a higher compressive

strength. Cubes 1, 4 and 5 from Batch C had deteriorated to such a degree during the freeze-thaw testing programme that they were unable to be compression tested and this halved the available cubes to provide data. The result of this test supports the theory that when rubber crumb is added to concrete, it provides freeze-thaw protection.

4.3 Pulse Velocity

The results from the pulse velocity testing to BS EN 12504-4:2004 are shown in Figure 7 and measurements were recorded after every 7 freeze/thaw cycles.

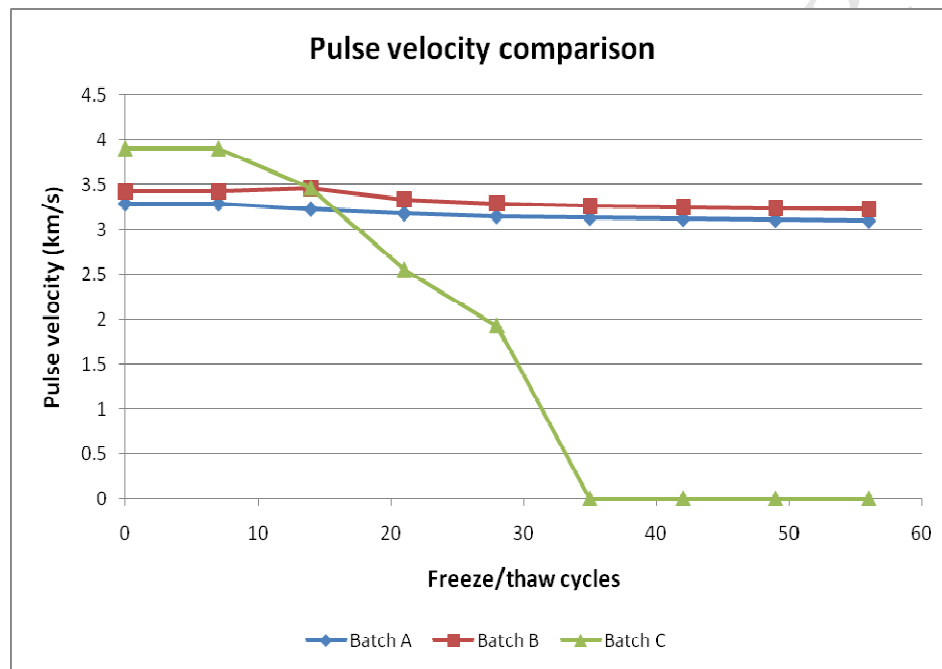


Figure 7 - Comparison of pulse velocity over the 56 cycle freeze-thaw programme.

Of all the specimens exposed to the freeze-thaw procedure, Batch B performed the best over the programme duration however this was only slightly better than Batch A. Overall, the rubberised concrete outperformed the plain concrete specimens, which showed a failure of 60% reduction in pulse velocity in all cubes after 28 freeze-thaw cycles.

4.4 Durability factor and weight lost

The durability factor was determined at the end of the freeze-thaw test and is shown in Table 4. A higher durability factor indicates enhanced freeze-thaw resistance.

	Durability Factor (%)
Batch A	96.4
Batch B	96.9
Batch C	18.85

Table 4 – Durability factor results

It is evident from the results of this durability investigation that the rubberised concrete obtained a much higher durability factor which can only be attributed to the rubber crumb addition. Figure 8 illustrates the weight loss suffered by batches A, B and C and the relative density of the concrete.

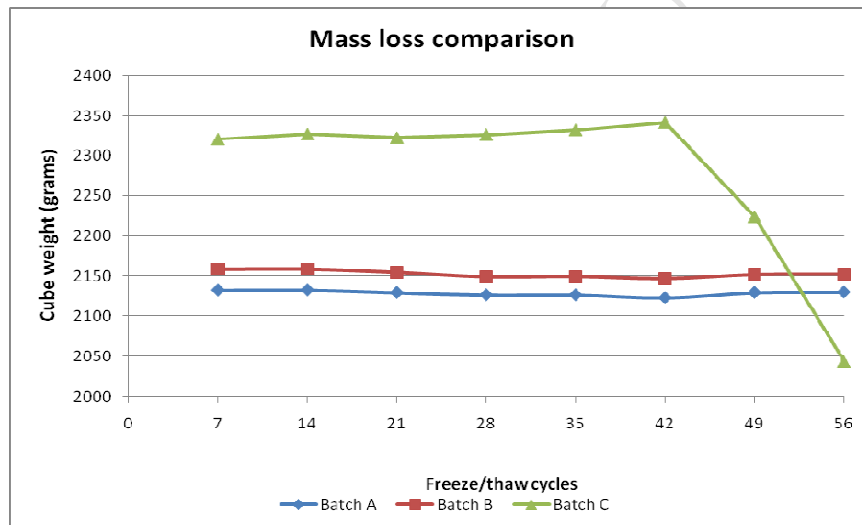


Figure 8 – Cube weight loss comparison by batch over successive freeze/thaw cycles

Batch A and B suffered minimal weight loss throughout the duration of the freeze-thaw process and any weight loss could be attributed to the slight scaling. Batch C showed an increase in weight then severe loss between freeze-thaw cycle 49 and 56. The slight increase in weight at cycles 35 to 42 could be related to the propagation of internal voids which filled with water.

The breakdown of the plain concrete cubes is shown in Figure 9.



Figure 9 – Batch C (plain) final extent of cube degradation

Figure 10 displays the intact and virtually damage free condition of the rubber crumb concrete cubes, which is representative of both rubber crumb samples.



Figure 10 – Batch B (unwashed rubber) final extent of scaling

4.5 Rubber particle distribution

Richardson et al. (2010) found that when compaction was carried out with a vibrating table the rubber crumb rose to the surface when vibration/compaction took place; which is not surprising as the specific gravity for ground rubber may be in the region of 480 kg/m^3 when compared with concrete which is 2400 kg/m^3 . This led to a surface laitance of rubber crumb which separated from the concrete. To investigate distribution of the rubber crumb particles, the cube was cut centrally with a water cooled masonry saw (to avoid melting the rubber particles) to show the rubber particle distribution within the concrete as shown in Figure 11.



Figure 11 – Rubber particle distribution

The hand tamping compaction, as used in this test, avoided separation of the rubber crumb from the concrete (as seen in Figure 11) and it is clear to see the distribution of rubber crumb throughout Batch A and B is both even and random.

The benefit of using an air-entraining agent, results from its ability to entrain, within the matrix of a concrete, numerous air voids which can relieve the stress due to the hydraulic pressure from the freezing water. The size of bubbles depends to a large degree on the entraining process used. The voids are not all the same size, and range usually from $0.05 - 1.25 \text{ mm}$ (Palliere, 1994). The adequacy of air-entrainment can be estimated by the spacing factor. This factor is the maximum distance of any point in the cement paste from the periphery of a nearby air void. It is generally accepted that a spacing factor of approximately $250 \text{ }\mu\text{m}$ is required to ensure that concrete in a severe freezing environment will be well protected. The rubber crumb dispersion is even and the upper pore size satisfactory when

compared to the accepted method of providing freeze/thaw protection using air entrainment in concrete.

5.0 Conclusion

The use of 0.6% rubber crumb by weight provided significant freeze-thaw protection in the concrete test specimens used for this study. The plain concrete samples failed before the completion of the freeze-thaw test programme whilst the rubberised samples had minimal surface scaling or internal damage. In addition to this, both rubberised concrete batches displayed a reduced overall density. This would further indicate the presence of internal air voids. Analysis of the rubber sample under a microscope showed how the rubber particles were irregular in shape and had a sharp jagged surface. This could explain how air is trapped due to the particle surface and shape and suggests that rubber crumb does in fact entrain air.

The use of rubber crumb in concrete has sustainable credentials in that it uses a waste product to enhance the performance of concrete and provide a material that will be more durable than plain concrete with subsequent lower life cycle costs due to reduced maintenance requirements. Using rubber crumb in this way is a much more cost effective way of utilisation, than simply burning in kilns for cement production and has few of the particulate emission problems associated with burning tyres.

Rubber crumb has been found to be effective in providing enhanced durability and if adopted as a concrete additive, the reduction in maintenance and remedial work to concrete may be significant.

6.0 Further work

The results show that a waste material can be incorporated into the supply chain and can support sustainable construction practices (Pelisser 2011). The challenge as described by Kürzinger (2004) is to build in the capacity of profitable environmental management. Small to medium sized businesses (SME) would be a good starting point as multinational companies have a much greater inertia to change. Paine and Dhir (2010) reiterate the need for the construction industry to develop rubber concrete products. repair sector is estimated to exceed 3% of the entire construction industry (Waterman 2006).

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