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Spring 2017

Rubber Additives to Concrete Specimens

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Sweitzer, Kendall J. and McCannon, Mary, "Rubber Additives to Concrete Specimens" (2017). *Honors Research Projects*. 555. [http://ideaexchange.uakron.edu/honors_research_projects/555](http://ideaexchange.uakron.edu/honors_research_projects/555?utm_source=ideaexchange.uakron.edu%2Fhonors_research_projects%2F555&utm_medium=PDF&utm_campaign=PDFCoverPages)

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Rubber Additives to Concrete Specimens

Kendall Sweitzer

12 May 2017

Table of Contents

Contents

Tables

Figures

Abstract

Rubber, as an additive to concrete, would hypothetically cause a concrete specimen to take on some mechanical characteristics of the rubber to a certain degree. In particular, the concrete's durability should increase when exposed to fluctuating temperature conditions due to the rubber additive. This experiment sets out to test crumb rubber as a concrete additive, cured under various atmospheric conditions. The effects shall be measured via a simple concrete compression test.

Unfortunately, several errors took place during experimental process that led to inconclusive results. However, it can be reasonably considered from testing Matrix One that the addition of crumb rubber does show a minor increase the durability of concrete in a compression test by approximately 5% when compared to the control samples. However, this was at the cost of approximately 50% of the compressive strength of the specimen. Testing Matrix Two also showed a drop in compressive strength by about 20%, but had other errors that made it difficult to draw any conclusion from. Finally, several possible hypotheses are discussed as to why these errors in testing may have occurred, though these hypotheses are also inconclusive without further research and testing.

Background

Durability in concrete is a highly desirable quality; high durability permits concrete to resist weathering and abrasion for longevity. One of the most common elements that plays a role in longevity is fluctuations in temperature, which causes concrete to contract and expand, decreasing the concrete's strength. Rubber, having a much higher coefficient of thermal expansion than concrete, would more readily expand and contract under heating and cooling

conditions and, therefore, may provide some benefit to the concrete's durability by causing less stress to accumulate in the specimen.

Past research has been conducted to put rubber in Portland cement concrete. Doing so produces several beneficial properties in the concrete mix. For instance, the addition of crumb rubber to concrete mixes helps decrease the unit weight of the material. Crumb rubber concrete is also more resilient to thermal changes relative to regular concrete mixes. It is more ductile than regular concrete and will better absorb mechanical energy. However, in crumb rubber concrete, flexural, tensile and compressive strengths all decreased as a result of the addition of $rubber¹$.

Using rubber as an additive to concrete has drawbacks beyond the strength reduction. It is believed that rubber is hydrophobic by nature (has a low wettability) and will repel water. Consequently, the cement-water paste will have little to no interfacial bonding with the rubber, and compressive strength will be lost due to this bonding deficiency within the sample². Hypothetically, by creating a coating or buffer between the rubber particles and the cement paste, this drawback may be lessened, resulting in a stronger concrete specimen. Prior research has been done to modify the rubber surface, making the rubber particles more hydrophilic (more wettable) and increasing bonding between the rubber and the cement paste³.

The wettability of a solid is determined by the angle that a liquid forms when it meets a solid surface. It is also depends on the interfacial tension between the solid-liquid, solid-vapor and liquid-vapor phases⁴. In essence, greater wettability of a substance allows more interfacial bonding with water to occur.

In a previous study, a three-step procedure was used for modifying crumb rubber. In it, crumb rubber was soaked in 5% sodium hydroxide for 24 hours, rinsed with water, soaked and

heated in 5% potassium permanganate at 60°C for 2 hours while keeping the pH around 2-3. Then, the rubber was rinsed with water and soaked and heated in saturated sodium bisulfite at 60° C for 0.5-1 hours. The study reported success in increasing the wettability of the rubber⁵.

In a second study, the performance of crumb rubber in concrete was tested when rubber was oxidized in a solution of potassium permanganate and then sulfonated in a solution of sodium bisulfite. The claim is that this method adds carbonyl, hydroxyl and sulfonate groups to the rubber surface, as indicated by FT-IR spectra of the untreated and treated rubber⁵.

To further substantiate the argument made in this second study, the contact angle was used to measure the degree of hydrophilicity (or wettability) of the rubber surface based on the change of the contact angle before treatment, after oxidation, and after sulfonation. Here, contact angle was measured with a HARKE-SPCA Video Optical Contact Angle Measurement and tests were run on rubber blocks.

Results show that rubber becomes more hydrophilic and that adhesive strength between the rubber and cement paste improves after treatment. This was verified by cutting small pieces from the rubber blocks, attaching them to a brick of cement paste as 10% of the total volume, and allowed to cure for 28 days. Each rubber piece was attached to a wire, which in turn was attached to a barrel. Adhesive strength was measured by filling the barrel with rock and sand until the rubber piece was pulled from the mold⁵.

Procedure and Methodology

Following this premise of these experiments and other research discussed above, an experimental scope is developed to explore for this project. The concept that is to be tested is how concrete specimens with a crumb rubber additive will react under various atmospheric

conditions. After a 28-day curing cycle, samples shall cure an additional 28 days at room temperature, at 4 °C, and at a temperature variation using recommendations from ASTM C666⁶.

The first concrete mixture devoid of additive, denoted as regular concrete, will serve as a control group to the additive samples. One of the additive sample groups shall consist of the raw, unaltered rubber and will serve as a control group to an altered rubber, with an increased wettability, inside the sample. The details of the alteration shall be discussed later on.

Upon creating the mix design for this matrix, it was discovered that not enough altered rubber could be obtained to make a full set of cylinders. Therefore, two sets of altered rubber additive were cut from the testing matrix. The curing room sample was kept as a control group. In addition, the set cured at a constant 20 $\rm{^{\circ}C}$ (cooled condition) was kept to see the effects of constant temperature and the freeze-thaw cycle group was kept because it provided the most dramatic changes in atmospheric condition. Table 1 shows the intended design matrix.

Set:	Regular Concrete	Unaltered Rubber	Altered Rubber
28 Day Break			
Curing Room			
Room Temperature			
Cooled Conditioning			
Freeze-Thaw Cycling			

Table **1:** Testing Matrix One showing the amount of cylinders made for each set

In this experiment, the rubber additive is in the form of crumb rubber because its small size can easily be distributed homogenously throughout a given concrete sample. The altered rubber set will then be modified in a 3-step process as described in the study by He et al.⁵. The crumb rubber shall be soaked in sodium hydroxide, potassium permanganate and then saturated sodium bisulfite. For the purposes of this paper, the effectiveness of this process shall be

measured using hydrophobic partitioning to indicate changes in the wettability of the two variations of rubber.

In this qualitative test, for each rubber sample, a vial is created with 5 mL of deionized water and 5 mL of hexane solution. Trace amounts of the rubber (approximately 0.1 grams) are then added and shaken vigorously. The distribution of the rubber will determine the wettability of the rubber. A hydrophobic material will disperse and stay suspended in the water, but will remain separated from the hexane. Conversely, a hydrophilic substance will disperse itself through the hexane solution, but be repelled by the water.

Mix designs for the specimens will be calculated using the weight and absolute volume method ⁷. For coarse aggregate, a #8 limestone was chosen to provide a higher strength to the mix and provide a larger contrast for analyzing results. A clean construction sand was chosen as an estimate of the specific gravity would be relatively accurate.

In addition to this standard baseline mix, rubber additive shall be added to the appropriate samples so as to make up 10% of a specimen by volume. This value was chosen with respect to past research, as briefly described above⁵. It is believed that such an amount will yield changes in the results without overtaking the entire concrete specimen. The full calculations for this mix design can be found in Figures 10-12 in the Appendix, but the results of the calculations are shown in Table 2.

	Regular	Unaltered	Altered
	Concrete	Rubber	Rubber
Water (g)	168.8	168.8	168.8
Cement (g)	383.7	383.7	383.7
Coarse Aggregate			
(g)	729.3	729.3	729.3
Fine Aggregate (g)	237.6	158.3	158.3
Rubber Additive			
g,	$0.0\,$	79.2	79.2

Table 2: Calculated proportion results for Matrix One for one cylinder

Concrete specimens will be made per ASTM C31⁸ standards and allowed to cure in a moisture-controlled room for 28 days. There is a general consensus that this length of time is appropriate because the majority of the cement will have had time to hydrate⁷. This ensures that the hydration process is a relative non-factor when comparing the compressive strength of the samples. After curing for 28 days, the cylinder sets would be separated to their various atmospheric conditions to cure for an additional 28 days.

However, upon stripping these samples 24 hours after preparation, a critical issue was discovered. Although all recommendations of ASTM C31 were correctly followed, concrete specimens exhibited various levels of honeycombing. It was noted upon providing compaction and consolidation to these samples that the large aggregate size, with a nominal size of $\frac{1}{2}$ inch, coupled with the small 3 inch x 6 inch cylinder mold left very large gaps between the large aggregate.

It is possible that the honeycombing was due to the rodding not adequately penetrating the sample layers and consequently not providing the correct degree of compaction. A second possibility is due to the ASTM standard not specifying instructions for 3 inch x 6 inch cylinder molds. This combination of nominal aggregate size and mold size may not be recommended for this very reason. In either event, if this set was to be recreated, a vibration table should have been employed to prevent this from occurring.

Due to the presence of significant honeycombing, these samples were inadequate for testing; however, for the purposes of this paper, the experiment would proceed on the matrix. In an attempt minimize the effect of this issue, samples would be divided so that each set would have, by inspection, as close to a homogenous sample representation as possible in respect to the honeycombing. An example of one of these sets is shown in Figure 1. It was further decided that, since the results of these sets are not reliable, a second batch of cylinders shall be made to expand the testing matrix and compare the results of the first mix design.

Figure 1: An example of a homogenous cylinder set with various levels of honeycombing

For this second matrix, it was decided that a finer course aggregate, a #57 Limestone, would be used to avoid the compaction issue. Also, with only trace amounts of altered rubber available, altered rubber samples were not considered for this new matrix. Table 3 shows the second intended design matrix to support the first one. Table 4 shows the new calculated proportion for matrix two for a single cylinder. Again, Figures 12-15 in the appendix show the full calculations for the mix design.

Table 3: Testing Matrix Two snowing the amount of cylinders made for each set			
Set:	Regular Concrete	Unaltered Rubber	
28 Day Break			
Curing Room			
Room Temperature			
Cooled Conditioning			
Freeze-Thaw Cycling			

Table 3: Testing Matrix Two showing the amount of cylinders made for each set

	Regular Concrete	Unaltered Rubber
Water (g)	384.2	384.2
Cement (g)	169.0	169.0
Coarse Aggregate (g)	759.9	759.9
Fine Aggregate (g)	270.1	85.5
Rubber Additive (g)	\mathcal{L}	79 2

Table 4: Calculated proportion results for Matrix Two for one cylinder

Immediately following the 28 day curing cycle for both of these matrixes, based on the recommendation from ASTM C666, freeze-thaw testing for the selected specimens will proceed. In this test, concrete specimens are rapidly frozen to $-18 \degree C$ over a two hour period and remain at that temperature for an additional 22 hours. After this 24 hour cycle, the specimens would then be thawed to 4 $\rm{°C}$ over two hours and then remain there over the next 22 hours under carefully controlled conditions. This process may be repeated for up to 36 cycles⁶. For the purposes of this experiment, 14 cycles were executed so that the strength of all cylinders could be tested for the 56 day compressive strength, an industry standard.

All specimens shall then be tested for their compressive strength using ASTM C39. For this experiment, ASTM C1231 unbounded rubber caps were used and, per the lab technician's training and request, specimens were loaded at $30,000$ lb/min \pm 5,000 lb/min⁹. Since the test does not specify loading for 3 inch x 6 inch cylinder, this value was taken proportionally from the 4 inch x 8 inch cylinder recommendations as an appropriate rate of advancement.

Results

Before results regarding the concrete samples may be discussed, observation from the Hydrophobic Partitioning Test must be observed. As shown in Figure 2, the middle vial containing unaltered rubber, crumb particles are repelled by the water and remain suspended in the hexane solution, proving that this crumb rubber, by nature, is hydrophobic. In comparison,

the vial on the far left, containing the altered rubber, shows particles suspended in both the hexane solution and partially in the water. In terms of wettability, the altered rubber is clearly still hydrophobic, but slightly less so than it was before. The vial on the far right contains a crumb rubber treated with NAOH, but is not relevant to this study. With more time, a more precise measurement of wettability could be conducted to further support these findings.

Figure 2: Resulting vials from hydrophobic partitioning shortly after being shaken

The full results of the experimental specimens are shown in Tables 7 and 8 in the Appendix. It should be noted that, per ASTM C39, only three cylinders are needed in order to establish a solid data point. By conservatively using recommendations for tolerance for 4 inch x 8 inch cylinders, anomalies in each set shall be discounted in calculations and analysis. As dictated by ASTM C39, if one of the cylinders was greater than a 10.6% difference in strength, it was discounted from the average as an outlier point⁹. Tables 5 and 6 display the calculated averages found for both matrices.

Set:	Unaltered	Unaltered	Altered
	Concrete (psi)	Rubber (psi)	Rubber (psi)
28 Day Break	7134	4366	
Curing Room	7819	4613	4153
Room Temperature	7470	4507	
Cooled Conditioning	7259	4492	4360
Freeze-Thaw Cycling	6925	4625	4346

Table 5: Modified averaged compression results from Matrix One

Set:	Regular Concrete (psi)	Unaltered Rubber (psi)
28 Day Break	7882	4189
Curing Room	2379	2010
Room Temperature	2428	1565
Cooled Conditioning	3083	1480
Freeze-Thaw Cycling	1850	1512

Table 6: Modified averaged compression results from Matrix Two

Figures 3-8 are graphical distributions of all strength data, presented by matrix and set. The black line across each of the data set marks the average for the data. It should also be noted that in Matrix Two, the data for the 28-day strengths were too high to view with the rest of the matrix's data and are shown separately in Figure 6.

Figure 3: Graphical representation of results for Matrix One, Regular concrete

▲ 28 Day Break ▲ Curing Room ▲ Room Tempurature ▲ Cooled Conditioning ▲ Freeze-Thaw Cycling

Figure 4: Graphical representation of results for Matrix One, Unaltered rubber concrete

Figure 5: Graphical representation of results for Matrix One, Altered rubber concrete

Figure 7: Graphical representation of results for Matrix Two, Regular concrete

Discussion

Despite the various levels of honeycombing that were present in the Matrix One specimens, there was little impact on the overall results. By placing the samples into relatively homogenous groups based on the severity of honeycombing, and then removing the outlier values that exceeded the tolerance in ASTM C39, an acceptable range of values was established. However, the simple existence of honeycombing in the samples disqualifies the samples from credible testing and the following conclusions drawn from this data should be considered preliminary.

A second issue regarding Matrix One was discovered, further discounting the adequacy of its results. The amount of fine aggregate in the concrete was incorrectly calculated for this batch. From Table 2 above, the amount of rubber added to each sample was subtracted from the total amount of fine aggregate by weight (instead of volume) to find the amount of fine aggregate

to include. As a result, more than 300% of the proper amount of fine aggregate was added, reducing the 10% proportion of rubber that was intended.

Despite this calculation error affecting the proportions of the mix, each set within Matrix One still remains homogenous and can be compared as such. To begin with, as predicted, the addition of a hydrophobic substance greatly decreases interfacial bonding within the cement paste, causing the strength of the specimen to decrease by approximately 40%. Curiously, however, the addition of modified rubber, on average, further decreased the strength in the concrete specimen by a minor 2% beyond the reduction from the unmodified rubber.

This phenomenon may partially be due to the severity of the honeycombing in these sets, but also suggests that the surface treatment of the unmodified rubber did very little to increase the wettability of the crumb rubber. This conclusion is further strengthened by the results of the hydrophobic partitioning. While it was clear from the results that contact angle was increased, the change was minor and negligible in terms of the original rubber sample.

However, some general strength trends can be derived from these results. From Figures 3 and 4, the 28-day strength results were among the lowest of the compression strengths. This is obviously due to the fact these samples only had half the time to strengthen their interfacial bonds compared to the other samples. The cooled conditioned sets were the next strongest due to the increase in curing time, despite being hindered from curing by the lower temperature retarding the reaction.

The cylinder sets kept at room temperature were, naturally, stronger still in the presence of a warmer atmosphere and saw an increase of about 4% in compressive strength comparably. As expected, the cylinders remaining in the curing room at an ideal curing conditions, proved to have the greatest compression strength, with a 10% increase compared to the 28 day results. This

is a testament as to why ASTM C31 calls for these atmospheric conditions, as they provide the most ideal conditions for the strength of the concrete, as opposed to having samples cure in the open air or at lower temperatures.

However, an exception to this general trend of increased strength over time can be found in Figure 3, the compression strength results for regular concrete. Under freeze-thaw conditions, the average strength result was similar to the 28 day break results with a 4% decrease. This trend suggests that under freeze-thaw conditions, the curing process greatly slowed. Furthermore, the constant fluctuation in temperature causing expansion and contraction in the concrete may further weaken the cement bonds within it.

In contrast, the unaltered rubber results in Figures 4 and 5 show that the freeze thaw compression result average is actually 5% higher than the 28-day strength, and is also roughly as strong as the 56-day curing room results. This suggests that, while the curing process was halted in these samples too, the addition of the rubber additive increased the durability of the sample as is expanded and contracted under freeze-thaw conditions. The unaltered rubber proved to have a much greater strength than its regular concrete counterparts found in Figure 3.

A similar conclusion can be drawn from Figure 5, showing the compression results of the altered rubber. In this case, the cooled conditions and freeze-thaw condition averages were quite close to one another, and proved to be approximately 5% stronger than the curing room samples. Both cases suggest that while strength was greatly weakened in compression with the addition of crumb rubber, minor improvements in atmospheric durability were achieved.

Although initially created as a confirmation to Matrix One, Matrix Two proved also to have a critical error, causing the compression results to be questioned as well. Although the 28 day results showed similar values to the results from Matrix One, all of the other sets broken

after 56 days showed drastic decreases in compressive strength. An issue of this magnitude would normally require additional research and theory to be understood. However, for the purposes of this study, one possible cause will be discussed below.

Figure 9 shows common examples of the types of breaks that were found in the 56-day tests for Matrix Two. These and many of the other samples broken that day exhibited vertical cracking. This may suggest that tensile stresses that developed perpendicular to the applied compression caused the sample to fail before the compressive strength capacity of the sample

was reached.

Figure 9: Common break results from the 56-day strength sets in Matrix Two

This may possibly be due to the end caps used in the experiment. Per ASTM C1231, unbonded caps may not exceed 100 cylinder breaks before being changed out¹⁰. Doing so may cause unsatisfactory results due to deformation in the cap. In an experiment conducted focusing on the use of hourglass-shape cylinder breaks for testing compression, it was noted that a decrease in friction between the plates causes less horizontal shear force to occur, resulting in vertical cracking¹¹.

One of the possibilities is that since the changing of the testing pads at The University of Akron is not monitored, the pads exceeded their maximum number of tests (per ASTM C1231). Doing so may have caused the pads to lose friction on the ends of the cylinder, causing this

vertical failure to occur. If this experiment was conducted again, bonded sulfur caps would be employed to ensure that then endcaps are in compliance with this test.

Due to the potential deficiency of the end caps used in this experiment, the compression strength of the Matrix Two data may have been drastically smaller than the actual compressive strength of these samples. This would explain why the compressive strength of the 28 day break results is so much higher than the 56 day results. Regardless of the reason, it was decided that the results between the 28 day and 56 day compression testing could not be compared.

Furthermore, this drop in compressive strength makes percentage variation in the data more dramatic and, therefore, more unreliable. For example, for the unaltered rubber set, Figure 8 and Table 6 show that there is nearly a 20% drop in compressive strength from the 28-day tests to the 56-day tests. At this stage, it cannot be deemed whether this is an accurate representation of the specimens.

Although the values obtained are much more precise than in Matrix One (with smaller differences in the strengths of the individual cylinders), the general trends do not seem hold true for the regular concrete in Figure 7 and may be the result of this error. However, the results still show a general drop in compressive strength in the freeze-thaw set, as previously discussed.

In retrospect, it is quite clear to see that small unaccounted-for errors early in the process ended up having significant detrimental consequences further along in the process. Although general trends did suggest that crumb rubber as an additive may have a positive effect on the durability of concrete, the errors in the data are far too numerous to say for certain. Furthermore, the general trends discussed should also be questioned as they are founded on only minor variations between different set types. In order to further substrate these hypotheses, a new, larger scope of testing would need to be created and evaluated.

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Appendix

*Value was removed from the calculation of the averages as an outlier

*Value was removed from the calculation of the averages as an outlier

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Figure 10: Page one of the full calculation details for Matrix One's mix design **Figure 10:** Page one of the full calculation details for Matrix One's mix design

Figure 11: Page two of the full calculation details for Matrix One mix design

Figure 12: Page three of the full calculation details for Matrix One mix design

Figure 14: Page two of the full calculation details for Matrix Two mix design

Proposed Mix Design 2 Honors Project Kendall Sweitzer	
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$-water: (410)$ (0.8246) $(3.7e^{3})$ = 338.99	
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Figure 15: Page three of the full calculation details for Matrix Two mix design