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Conductive paint-filled cement paste sensor for accelerated percolation

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Abstract

Cementitious-based strain sensors can be used as robust monitoring systems for civil engineering applications, such as road pavements and historic structures. To enable large-scale deployments, the fillers used in creating a conductive material must be inexpensive and easy to mix homogeneously. Carbon black (CB) particles constitute a promising filler due to their low cost and ease of dispersion. However, a relatively high quantity of these particles needs to be mixed with cement in order to reach the percolation threshold. Such level may influence the physical properties of the cementitious material itself, such as compressive and tensile strengths. In this paper, we investigate the possibility of utilizing a polymer to create conductive chains of CB more quickly than in a cementitious-only medium. This way, while the resulting material would have a higher conductivity, the percolation threshold would be reached with fewer CB particles. Building on the principle that the percolation threshold provides great sensing sensitivity, it would be possible to fabricate sensors using less conducting particles. We present results from a preliminary investigation comparing the utilization of a conductive paint fabricated from a poly-Styrene-co-Ethylene-co-Butylene-co-Styrene (SEBS) polymer matrix and CB, and CB-only as fillers to create cementitious sensors. Preliminary results show that the percolation threshold can be attained with significantly less CB using the SEBS+CB mix. Also, the study of the strain sensing properties indicates that the SEBS+CB sensor has a strain sensitivity comparable to the one of a CB-only cementitious sensor when comparing specimens fabricated at their respective percolation thresholds.

Keywords

Cementitious sensor, carbon black, percolation, cement-paste sensor, SEBS, structural health monitoring

Disciplines

Civil Engineering | Controls and Control Theory | Electrical and Electronics | Polymer Science | Structural Engineering | VLSI and Circuits, Embedded and Hardware Systems

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Conductive Paint-Filled Cement Paste Sensor for Accelerated Percolation

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ABSTRACT

Cementitious-based strain sensors can be used as robust monitoring systems for civil engineering applications, such as road pavements and historic structures. To enable large-scale deployments, the fillers used in creating a conductive material must be inexpensive and easy to mix homogeneously. Carbon black (CB) particles constitute a promising filler due to their low cost and ease of dispersion. However, a relatively high quantity of these particles needs to be mixed with cement in order to reach the percolation threshold. Such level may influence the physical properties of the cementitious material itself, such as compressive and tensile strengths. In this paper, we investigate the possibility of utilizing a polymer to create conductive chains of CB more quickly than in a cementitious-only medium. This way, while the resulting material would have a higher conductivity, the percolation threshold would be reached with fewer CB particles. Building on the principle that the percolation threshold provides great sensing sensitivity, it would be possible to fabricate sensors using less conducting particles. We present results from a preliminary investigation comparing the utilization of a conductive paint fabricated from a poly-Styrene-co-Ethylene-co-Butylene-co-Styrene (SEBS) polymer matrix and CB, and CB-only as fillers to create cementitious sensors. Preliminary results show that the percolation threshold can be attained with significantly less CB using the SEBS+CB mix. Also, the study of the strain sensing properties indicates that the SEBS+CB sensor has a strain sensitivity comparable to the one of a CB-only cementitious sensor when comparing specimens fabricated at their respective percolation thresholds.

Keywords: Cementitious sensor; carbon black, percolation, cement-paste sensor, SEBS, structural health monitoring

1. INTRODUCTION

Structural health monitoring (SHM) of civil structures has the potential of enabling timely inspection and maintenance, resulting in enhanced structural safety and longer life span.[1,2] However, the SHM task is complicated by the inherent size of the structures to be monitored. Most of existing sensing solutions are hardly scalable without necessitating substantial costs and complex signal processing algorithms. It results that monitoring and diagnostic solutions may rapidly become financially unattractive because of their low return on investment.

Recent advances in nanomaterials and synthetic metals have led to new possibilities in sensor developments,[3,4] including new multifunctional materials that enable substantial improvements in the cost-effectiveness of SHM solutions for geometrically large systems.[5] In particular, sensors fabricated from cementitious-based nanocomposites have been proposed. The sensing principle is based on measuring a change in resistance provoked by a change in geometry and resistivity when the level of conductive particles is at the percolation threshold. Cementitious-based sensors have the potential to create large distributed network, can be easily embedded in traditional cementitious materials, and form a strong mechanical bond with the monitored structure. Several nanofillers have been studied to fabricate cementitious sensors, including carbon black (CB),[6] carbon fiber,[7] and carbon nanotubes (CNTs).[8]

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Conductive concrete has been studied and utilized for a few decades, primarily for de-icing of road infrastructures.[9–11] The development of conductive cementitious materials for sensing purposes is more complex, because the nanocomposite must be fabricated in a way to provide a linear and sensitive signal with respect to a change in the measured state. There are important challenges that limit the cost-effective fabrication of large volumes of such conductive cementitious materials, namely: 1) there is a trade-off between the cost of the selected conductive filler and its conductivity; and 2) the utilization of high conductivity nanoparticles, such as CNTs, leads to a complex fabrication process due to the difficult dispersion of the particles, which impedes scalability of the fabrication process.

In this paper, the authors propose to leverage a polymer to accelerate the percolation threshold of a cement paste using inexpensive conductive particles. The polymer is a poly-Styrene-co-Ethylene-co-Butylene-co-Styrene (SEBS) and the conductive filler is carbon black (CB). The authors have previously used SEBS+CB to fabricate a conductive paint for the conception of large-scale flexible strain gauges.[12,13] Here, this conductive paint is mixed within a cement paste to fabricate a cementitious sensor, termed conductive paint cement paste sensor (CPCPS). This paper presents results from a preliminary investigation of the percolation behavior of CPCPS in comparison to CB-based cementitious sensors.

The paper is organized as follows. Section 2 presents the methodology, including a description of materials and the fabrication process. Section 3 presents test results and compares the performance of a CPCPS against a cement-paste sensor. Section 4 concludes the paper.

2. BACKGROUND

This section provides a background on the materials selected in the fabrication of CPCPS, and on the fabrication process itself.

2.1 Materials

SEBS type Mediprene was obtained from VTC Elastoteknik AB, Sweden (density = 930 kg/m^3). It is a petroleum-based block copolymer widely used for medical applications, because of its purity, softness, elasticity, and strength.[14] CB type Printex XE-2B (2% ash content and 500 ppm sieve residue 45 μ m) was acquired from Orion Engineered Carbons (Kingswood, TX). It is characterized by a high structure (minimum oil absorption 380 cc/100g), which facilitates higher conductivity.[15] Copper meshes to form the electrodes were acquired from McMaster-Carr (Elmhurst, IL). Portland cement type I/II was locally purchased from Ash Grove Cement Company.

2.2 Fabrication Process

The fabrication process of a CPCPS is illustrated in Fig. 1. SEBS is dissolved in 100 ml of toluene per 30 g of SEBS for 12 hours (Fig. 1(a)). CB particles are added to the solution and dispersed in a sonication bath for five hours (Fig. 1(b)). The resulting solution is added to a volume of water corresponding to a water-to-cement (w/c) ratio of 0.45, and dispersed using a surfactant (sodium lauryl sulfate or SLS) in a sonication bath for five hours (Fig. 1(c)). Portland cement is added to the solution and manually mixed for 5 minutes. A plasticizer (Glenium 7500) is added in increments of 1 ml until satisfactory workability while mixed using a high shear mixer (Fig. 1(d)). This volume of plasticizer increases with the amount of CB used in the mix due to higher water adsorption. The filled cement-paste is then poured in a $2 \times 2 \times 2$ in^3 mold (Fig. 1(e)) and copper mesh electrodes inserted at two opposite edges (Fig. 1(f)). The cement paste was let dry at room temperature for 1 day, demolded, and cured in water for 3 additional day (Fig. 1(g)). Blocks are dried in an oven at 50°C for 3 days to let the water evaporate. A similar procedure is used to fabricate the CB-based cementitious sensors, where CB is directly dispersed in water using a sonication bath, without surfactant.

Table 3 lists the 20 different mixes under study. Mix 1 is the control mix (pure cement paste). Mixes 2-10 do not contain SEBS. Mix 11 is the SEBS control specimen (SEBS-cement paste only), and mixes 12-20 contain SEBS+CB (or CPCPS). Three specimens per mix were fabricated, for a total of 60 specimens.

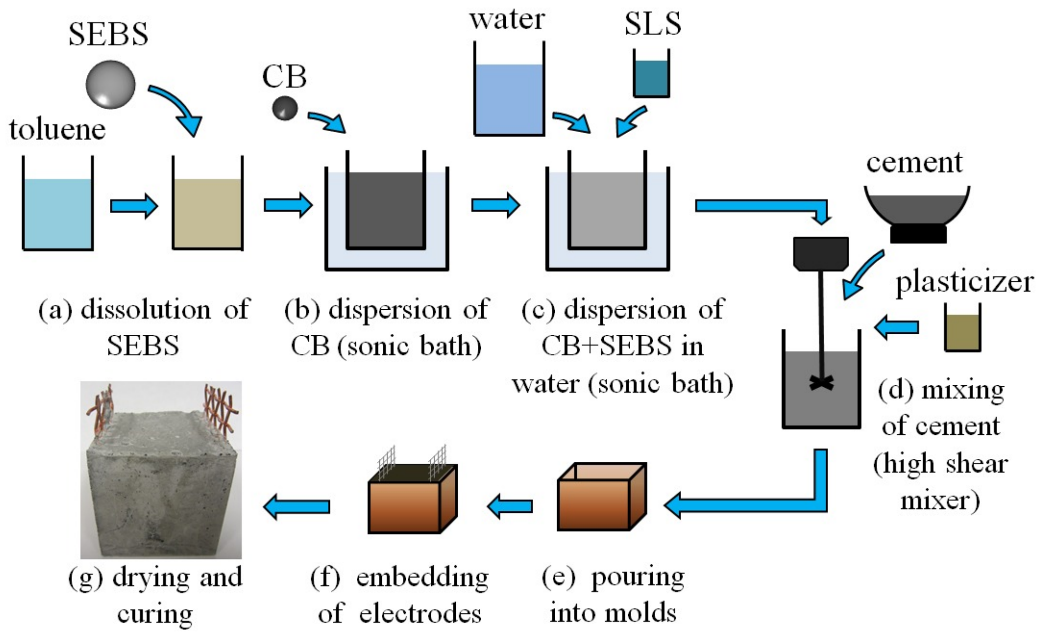


Figure 1: Fabrication process of a CPCPS.

Table 1: Mixes under study

mix #	CB vol%	CB (g)	SEBS (g)	cement (g)	water (ml)
1	0.00	0.00	0.00	213.5	96.1
2	0.20	0.62	0.00	212.7	96.0
3	0.31	0.96	0.00	212.7	96.0
4	0.41	1.27	0.00	211.9	95.9
5	0.82	2.53	0.00	210.3	95.8
6	1.61	4.97	0.00	210.3	95.5
7	2.37	7.35	0.00	204.2	95.2
8	3.12	9.66	0.00	201.2	94.9
9	3.85	11.9	0.00	198.3	94.6
10	4.61	14.3	0.00	198.3	94.3
11	0.00	0.00	3.26	195.3	87.9
12	0.20	0.62	3.26	195.3	87.1
13	0.31	0.96	3.26	195.3	86.9
14	0.41	1.27	3.26	193.7	87.3
15	0.61	1.89	4.89	193.7	87.0
16	0.66	2.02	5.22	189.0	85.0
17	0.71	2.21	5.71	187.4	84.0
18	0.82	2.53	6.55	173.3	78.6
19	1.20	3.75	9.70	155.9	69.6
20	1.61	5.05	13.1	135.5	60.6

3. RESULTS

This section presents results from this preliminary study. First, the percolation threshold of the CB mixes (mixes 1-10) is compared against the percolation threshold of the SEBS-CB mixes (mixes 11-20), in order to verify the hypothesis that the SEBS can be used to accelerate percolation. Then, specimens are selected among mixes, and tested as strain sensors. The objective of this second series of tests is to quantify the dispersion of the CB by investigating the linearity of the signal, and to compare the sensitivity of the sensors at and around the percolation thresholds.

3.1 Percolation Threshold

The resistivity of each sensor is measured using an Agilent 4263B LCR meter at 100 kHz under 1000 mV after the sample has been dried out in the oven and let cool down to room temperature. Fig. 2 shows the relative resistivity ρ of each sample as a function of weight % per volume of CB, where ρ_0 is the measured resistivity of the control sample (0 vol% CB). A percolation threshold between 0.82 vol% and 2.37 vol% CB was observed for the CB-only samples (Fig. 2(a)), while this threshold was between 0.61 vol% and 0.82 vol% CB for the CPCPS samples (Fig. 2(b)). Despite that the overall resistivity for the CPCPS samples past the percolation threshold is approximately an order of magnitude higher compared to CB-only samples (Fig. 2(a)), results confirmed the authors' hypothesis that a polymer could be used to accelerate percolation of a cementitious material, enabling the fabrication of sensors using a reduced loading of conductive particles. Note: the quantity of SEBS used in each specimen varied as a function of the quantity of CB; the objective was to use the smallest quantity of SEBS possible while fabricating an un-saturated CB+SEBS mix.

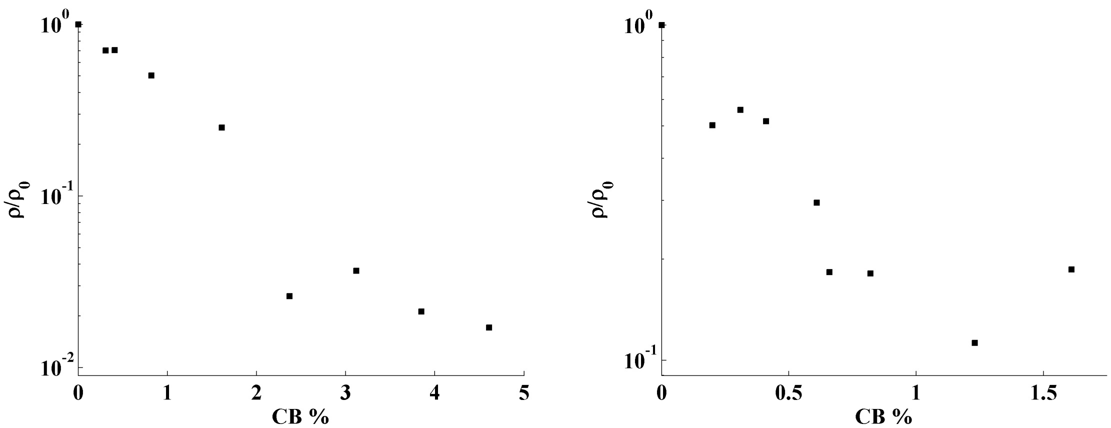


Figure 2: Relative resistivity as a function of CB content (in % of total volume): (a) CB-only; and (b) CPCPS.

Preliminary compressive strength tests were conducted on the cement paste as a function of the concentration level of CB. Samples were taken at both percolation thresholds observed in Fig. 2: one at 1.61 vol% CB (from CB-only - Fig. 2(a)) and one at 0.61 vol% CB (from CPCPS - Fig. 2(b)), along with control specimens at 0 vol% CB. Table 2 lists the compressive strengths at seven days of curing. While the samples at 0.61 vol% CB do not show a significant decrease in compressive strength, samples taken at 1.61 vol% CB exhibited a decrease of 26.0% in compressive strength. Note that the CB particles in this study were used as received. It would be possible to increase the compressive strength of the cement paste by pre-separating CB particles and utilizing a given particle size only.

Table 2: Maximum compressive strength at seven days

	0 vol% CB	0.61 vol% CB	1.61 vol% CB
average (psi)	7160	7000	5300
standard deviation (psi)	613	271	379
reduction in strength (%)		2.28	26.0

3.2 Strain Sensing

The strain sensing capability of the CPCPS was assessed in terms of linearity of the signal and sensitivity. Eight specimens were selected from the percolation threshold results, and tested as strain sensors. Four samples were CPCPS, and taken before the percolation threshold - 0.41 vol% CB (SEBS 0.41%) - at the percolation threshold 0.61 vol% CB (SEBS 0.61%) and 0.66 vol% CB (SEBS 0.66%) - and past the percolation threshold - 0.82 vol% CB (SEBS 0.82%). The four other samples were CB-only cementitious sensors used for comparison, and taken before the percolation threshold - 0.41 vol% CB (CB 0.41%), at the percolation threshold - 0.82 vol% CB (CB 0.82%) and 1.61 vol% CB (CB 1.61%) - and past the percolation threshold - 2.37 vol% CB (CB 2.37%). Three specimens were tested for each of the eight selected mix. Results presented in this section are from a representative specimen exhibiting a typical behavior.

Each sample was subjected to compressive strain using a 11.25 kips capacity Instron 5969 testing system. The applied strain load ranged from 0 to 1000 $\mu\varepsilon$ with increment steps of 100 $\mu\varepsilon$ ramping at a rate of 5 $\mu\varepsilon$/s. Load and strain from the Instron machine were recorded using the built-in DAQ. The impedance was manually recorded using an Agilent 4263B LCR meter at 100 kHz under 1000 mV. The test setup is shown in Fig. 3.

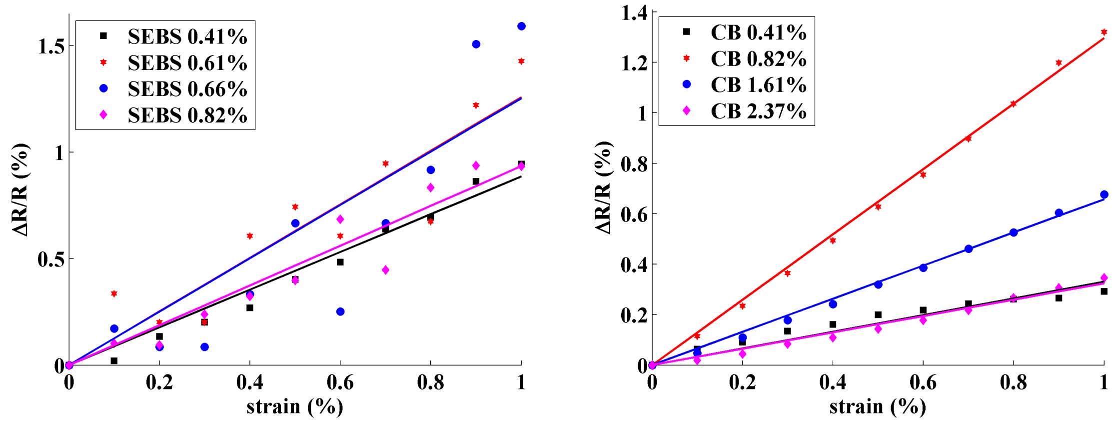
Figure 3: Setup for strain sensing tests.

Figure 4 compares the relative change in resistance for CPCPS (Fig. 4(a)) and CB-only sensors (Fig. 4(b)) as a function of the applied strain. The linear fit represents the sensitivity of the sensor's resistance with respect to strain. Sample SEBS 0.66% exhibits a higher level of noise. The samples at percolation (SEBS 0.61% and SEBS 0.66%) exhibit a higher sensitivity (the linear fit for both sensors overlaps). This result is consistent with literature,[16] which suggests that the sensitivity is maximal at the phase of transition. This also confirms the location of the percolation threshold. Results are similar for the CB-only sensors (Fig. 4(b)), but exhibit less noisy measurements. Sample CB 0.41% shows a slight nonlinearity.

Noise and nonlinearity in the signal can be explained by a nonuniform dispersion of the filler. Another explanation is the possible larger contribution of the dielectric of the material found in CPCPS and the CB 0.41% (lower loading of CB%). Fig. 5 compares the relative change in impedance for both mixes as a function of strain. A similar trend in terms of sensitivity is observed, except for a significantly higher sensitivity of samples with lower CB loading (SEBS 0.41% and CB 0.41%) with respect to samples beyond the percolation threshold (SEBS 0.82% and CB 2.37%). Moreover, CPCPS samples are less noisy, and the CB 0.41% sample appear to be linear. This confirms the contribution of the dielectric. It can also be observed that the sensitivity of the CPCPS in terms of relative change in impedance is significantly less than the one of CB-only sensors. This is the converse for the sensitivity in terms of relative change in resistance, except for the samples SEBS 0.61% and CB 0.82% that have a comparable sensitivity.

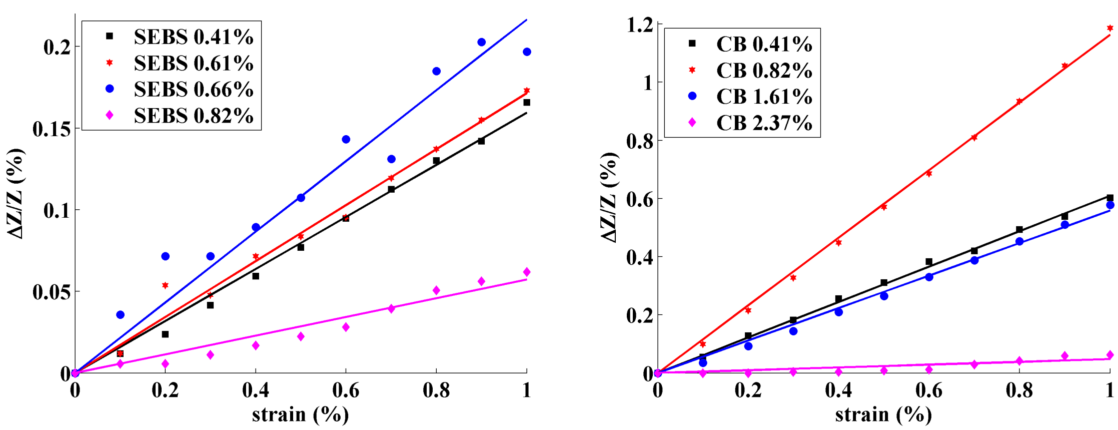
Table 3 lists the gauge factors for all eight samples in terms of relative resistance λ_R and impedance λ_Z corresponding the the slope of the linear fits. The highest gauge factor for both types of sensors is around

$\lambda_R = 1.3$, both obtained with the resistance signal around the percolation threshold. It would be possible to optimize this sensitivity to strain by further tuning the CB loadings. This is left to future work.



(a) (b)

Figure 4: Relative change in resistance versus strain: (a) CPCPS; and (b) CB-only.



(a) (b)

Figure 5: Relative change in impedance versus strain: (a) CPCPS; and (b) CB-only.

Table 3: Experimental gauge factors

sensor	CB (vol%)	λ_R	λ_Z
CPCPS	0.41	0.885	0.160
CPCPS	0.61	1.257	0.171
CPCPS	0.66	1.253	0.217
CPCPS	0.82	0.934	0.0572
CB	0.41	0.330	0.610
CB	0.82	1.300	1.164
CB	1.61	0.657	0.558
CB	2.37	0.324	0.0479

4. CONCLUSION

This paper investigated the utilization of a polymer, SEBS, to accelerate percolation of cement-paste filled with CB particles. Building on the principle that the percolation threshold provides great sensing sensitivity, it would therefore be possible to fabricate sensors using less conducting particles. Results from a preliminary study were presented. It was found that the percolation threshold of an SEBS+CB mix was located between concentration levels of 0.61 vol% and 0.82 vol% CB, while that of a CB-only mix was located between concentration levels of 0.82 vol% and 2.37 vol% CB . Thus, by using an SEBS to facilitate the creation of conductive CB chains, it was possible to create cementitious sensors (CPCPS) by significantly reducing the amount of CB particles. Such reduction in the amount of conductive particles could also significantly increase compressive strength of the material, as it was demonstrated with the compressive strength tests conducted on CB-only mixes. Strain sensing tests were conducted on CPCPS and benchmarked against CB-only cementitious sensors. Results confirmed the location of the percolation thresholds, and showed good linearity of the sensors.

These preliminary results and findings demonstrated that it is possible to fabricate large-scale conductive mixes for sensing purposes, utilizing only a small fraction of conducting particles, by integrating a polymer in the mix to facilitate the creation of conducting chains. Future work includes the optimization of the SEBS+CB mixing procedure to improve dispersion of the CB particles, therefore improving the sensor's linearity, and investigating the compressive and tensile strength properties of the CPCPS mixes.

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