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Autonomous cementitious materials formulation platform for critical infrastructure repair

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Novel cement formulations are critical for meeting the global need for infrastructure repair. Despite the long history of cement and concrete development and use, there are still large gaps in the fundamental knowledge of their properties, frustrating the design of new formulations. The dire need for improved cementitious repair materials, therefore, requires a revolution in the way they are formulated and tested. Here we suggest an autonomous platform that could be leveraged to rapidly formulate and test designed-for-purpose cement patch materials.

1 Introduction

While cementitious materials have been around for millennia, there are still grand societal challenges that can be met by advances in these materials. As the most utilized synthetic material globally, concrete's popularity for physical infrastructure applications (*e.g.*, buildings, roads, bridges, tunnels, *etc.*) can in-part be attributed to the "just-add-water" nature of most modern cement-based materials and their widely available, inexpensive mineral precursors. Despite the long-history of these materials, there still remains numerous challenges with regards to the fundamental (*e.g.*, thermo-kinetic[1,2] and rheological[3]) understanding of these materials, critical to efficient development and optimization of new formulations. In particular, there are two great societal needs that can be addressed by advances in construction materials: (1) the need to repair and replace deteriorating physical infrastructure; and (2) the need to reduce CO_2 emissions and atmospheric levels.

It is well documented that there is a large amount of deteriorating infrastructure worldwide. In the United States the American Society of Civil Engineers (ASCE) estimates an investment of 2.8 trillion USD is needed to repair or replace surface transportation infrastructure to satisfactory levels. This high cost is partly due to the fact that 7.5% of bridges nationally are deemed structurally deficient, though 178 million trips are taken over them daily.[4] In Europe, 35% of the over one million bridges in the railway network are over 100 years old as of 2015.[5] Further, 215 million GBP is spent per anum on maintenance and repair of the structural elements of concrete bridges by the European Union.[6] China, which has the most number of bridges in the world, has a higher proportion of structurally deficient bridges than the US.[5] Given the state of global infrastructure, improving concrete repair methods and materials has the potential to save both lives and money.

Environmentally, the production of ordinary Portland cement (OPC) accounts for approximately 8% of global generation of CO_2.[7] This is primarily due to the process of calcination that involves heating limestone to transform $CaCO_3$ to CaO to form clinker, the precursor to cement (both directly from the chemical reaction as well as from the energy required to heat the material). In addition to this CO_2 emitted directly for the production of OPC, there is also the emissions associated with the transportation of raw ingredients and deployment of cement and concrete at worksites. While there are ongoing efforts to reduce the amount of CO_2 released by cement based materials,[7,8] reducing the demand for cement can also reduce CO_2 emissions. To this end, repairing existing infrastructure rather than replacing it, in addition to the fiscal savings, can greatly reduce the volume of new material needed and the CO_2 emissions related to its production while increasing public safety.

While there is a clear need for infrastructure repair, cement for infrastructure repair is highly specialized and requires a reverse design process that is somewhat unique in the field of cementitious materials. Modern repair-work is often performed using alternative mixture formulations,[9–12] which typically harden and develop strength faster than standard OPC formulations; short curing times are important to minimize the down time of the infrastructure.[13] The repair material must also be compatible with the existing material; the repair material must be of a similar strength and have a similar coefficient of thermal expansion as the original material. The repair material must have minimal shrinkage associated with the curing process so

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that the material fully fills the repair cavity in the structure.[14] Further, areas of a given structure requiring repair can sometimes be difficult to access and challenging to repair for vertical or overhanging locations. Repair-work for these locations can require special considerations with respect to the cement that is used. That is, a given mixture depends on the application and low-viscosity and firmly bonding to the substrate structure are useful characteristics.[15]

Given this highly constrained problem, identifying appropriate formulations is a challenging task. However, because of the need for the repair material to match a particular host material, which will vary job-to-job, there is no universal optimal composition. Therefore, this is an ongoing design challenge. Complicating this design challenge is a lack of fundamental understanding and therefore predictive, physics based models. Further, the large number of constituents and their respective mixing ratios makes an Edisonian approach intractable. To this end, a machine learning based surrogate model to map input material to properties can be a valuable tool.[16] However, these models are only accurate in the neighborhood of existing data. Ideal formations for repair materials may be far from existing knowledge bases, to say nothing of novel components. Therefore, validation and targeted improvement of the model will require access to on-demand data generation. An autonomous platform is ideal for this type of dynamic data generation.

Here we describe our vision for an automated cementitious materials testing platform. This platform will consist of a synthesis tool for mixing and dispensing the material, as well as a set of tools for testing the curing and cured material. The overall system will be controlled by a set of autonomous machine learning (ML) agents that will direct an active learning feedback loop.

Before we delve into the details of our proposed autonomous system, we note a few differences between this system and many of the other material acceleration platforms currently being developed:[17] first, the methods in this space are well described by ASTM and other standards, some of which will be described in §4. Given the safety implications of the materials being developed, it would behoove the developer of such an autonomous system to follow these standards where possible in order to accelerate the time-to-market. Second, while some measurements take place on the uncured product, measurements occurring on the curing or cured product may have long timescales, particularly compared to the mixing and dispensing phase. Third, the cost and scale of the material is much different than many other autonomous systems. We will likely need to produce liters of material per sample, but this material is much cheaper than many other types of materials.

These unique material needs and testing requirements will inform the design of the autonomous system. For instance, given the low cost and long time horizon of preparation for some tests, it may be more economical, both temporally and fiscally, to produce specimens when a given mixture is being produced and then decide whether to actually run the test based on a later model updated with additional data. Further, this long time horizon may make it preferable to use technicians responsive to the ML agent, to run certain tests, that are costly to fully automate. To account for variability in the timescales for the material testing, we will employ an active learning strategy that weighs the desirability of obtaining the results of the measurement with the time cost of performing the measurement. This will work in conjunction with a constraint programming based experiment planner to allocate resources efficiently, enabling parallel experiment strategies.

2 Cement materials

As mentioned above, modern cementitious materials are constitutively complex. Portland type cements, most commonly used, begin with a hydraulic lime mixture typically also containing alumino-silicates. To this a variety of supplementary cementitious materials (SCMs) can be added to modify the properties and potentially reduce the cost of the mixture. Traditionally these materials, which take part in the hydration reaction during curing, include fly ash, slag, and silica fume though more recently other materials are being developed and used.[8] Admixtures can also be added to the cement paste to modify the properties of the mixture. These can include modifying the setting time, the viscosity, the durability, and the amount of water needed.[13] Admixtures are an area of ongoing research and development and new compounds are constantly being introduced. A special case of this is the development of polymer cements, where a polymer resin acts as a co-binder along with the traditional cement material. Epoxy resins are of particular interest, due to their high strength, fast setting time, good adhesion, and resistance to environmental degradation mechanisms.[18]

Hydration – a dissolution–precipitation process – of cement is one of the most critical steps in the process. The water-to-binder ratio is an important factor in the properties of the product and is an important variable in any cement study.

Finally, there are non-reactive materials that are added to cement paste. Aggregate is added to produce concrete and sand to make plaster. A newer development is the mixture of cement with polymer fibers to increase the strength and toughness of the cement. Due to the difficulty of handling aggregate we will mostly focus on cement and mortar in this work.

3 Automating synthesis

Perhaps the most important part of the platform described here is the ability to automate the mixing of the cement. We envision a batch mixing plant for the dry ingredients combined with a continuous mixer and pump for the hydrated cement/mortar as illustrated in Fig. 1. The mixing plant begins with a set of automated hopper-feeders, one for each dry ingredient to be used. The complexity of the material and scope of the region that can be explored in a given experimental campaign will scale with the number of hoppers. Dosing from these feeders can be pre-calibrated or for additional precision, each can be located on a scale to measure the amount of material that is dosed. These feeders supply material to a mixer which will mix the raw dried material. The material will then be loaded into a hopper that feeds a continuous mixer/pump. Here water along with any other liquid admixtures (dosed continuously with a peristaltic

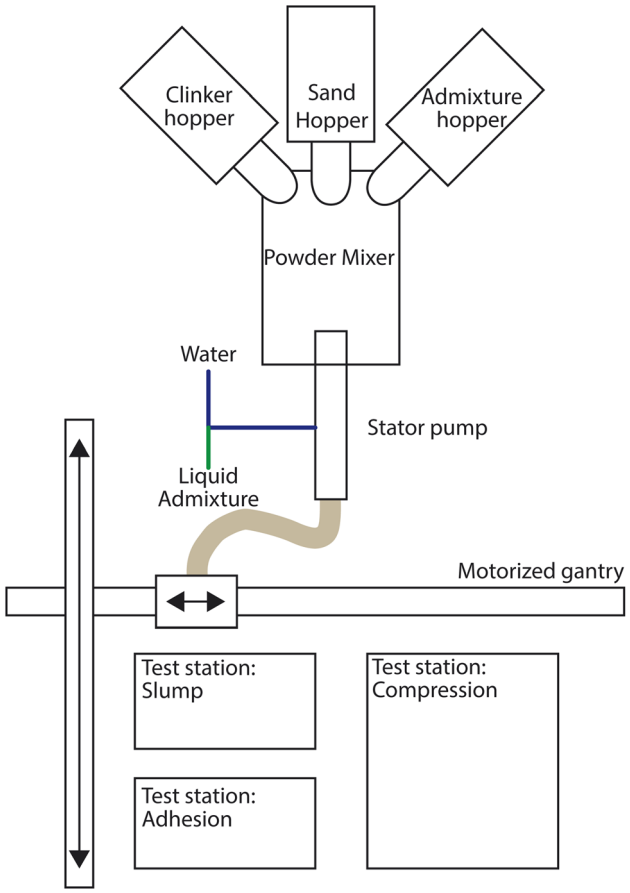

Fig. 1 Cartoon schematic showing the layout, viewed from above, of the described automated synthesis and characterization platform. System complexity can be scaled up or down depending on the scope of the research.

pump) will be combined with the dry powders to form the cement paste. In this configuration, the solid mixture will be fixed for each batch but the liquid additions can be changed sample-to-sample.

The pump will then push the cement paste, through a tube to a nozzle located on a gantry. The tube should be made as short as possible to reduce the amount of material that needs to be purged between samples. The gantry will be able to position the nozzle and supply cement paste to any of a number of experimental stations as will be described below. Liquid accelerators may be added at this point.[13] In addition to the nozzle, the carriage of the gantry may benefit from having two additional features: The first is an oil mister to provide oil which can act as a release agent for between the concrete and reusable receptacles. Second is a method of processing the cement in the molds. Many ASTM standard tests require consolidation of cement through tamping as well as leveling with a trowel. While in theory these actions could be performed by a technician working alongside the nozzle, mechanizing these actions will increase the consistency of the data. These items could also be integrated into the mechanism of each characterization station.

While the system will need to be carefully engineered and assembled, many of the base components mentioned above are commercially available. Further they are typically designed with industrial automation capabilities or have simple electrical controls that can be modified to include a programmable logic controller. This plant can likely make use of much of the technology that has been developed for cement based additive manufacturing and spray coating.[19]

The synthesis will be controlled by an ML agent as described in §5. There are three levels of decisions that need to be made, due to the timescale of the feedback loop, largely upfront. The agent first must decide on a solid mixture. Second, the agent can then decide what hydration ratio and other liquid admixtures will be used; these can be varied across a given solid mixture. For both of these decisions, the ML agent will consider the mixture space that has yet to be explored and which mixtures are predicted to be near the desired properties to determine the importance of the unmeasured mixtures and choose which mixtures to study next. Finally, for each product made, the agent must decide which tests to perform (or in the case of some tests, to prepare samples for). Here the ML agent will weigh the desirability of obtaining the results of the tests against the costs (time and material) of performing the measurement (and where applicable, the availability of the measurement apparatus) using an acquisition function such as upper confidence bound with a integrated cost function.[20] The machine can then add the required quantities across all the tests and hydration variations to determine the quantity of dry ingredients that need to be added to the dry mixer to start the process. In general, we expect each material to need on the order of 10s of liters of final material for testing, dosed out in a range of volumes for various testing protocols.

The largest challenge we expect with respect to synthesis relates to cleanliness. Care must be taken in the design of the instrument to avoid cross contamination of different products, both the powdered precursor mix as well as the hydrated material. The dry hoppers and mixers must be able to be fully emptied of all but trace amounts of powder. For the paste downstream of the hydration pump, the new material should force out the old material but this will have to be carefully calibrated. Again, here we note that due to the relative low cost of ingredients, it is reasonable to purge the system into waste to ensure the purity of the material. Another challenge will be managing the work time of the hydrated material; ensuring that hydrated material is used within a reasonable working time.

4 Automated characterization

Under the dispensing gantry can be a series of automated characterization stations. These stations can be fully automated or require human interaction, and each station can be self-contained and modular. Each station must be capable of generating a test specimen from the supplied material including consolidation, running the test, handling and storing samples as needed, and cleaning the apparatus. As mentioned above many of the necessary measurements are described by ASTM and other standards. However these standards are not inherently designed to be automated. Creating methods to automate these test will be another substantial engineering

challenge. In some cases this can be readily achieved, while in others modifications in method or scale may be required, and it will be necessary to demonstrate the results are consistent with those generated using standard methods. However, if this can be achieved, it will remove non-systematic bias from the results.

As mentioned above, the characterization methods can be broadly clustered into three categories based on where in the curing process they occur: on uncured paste, *in situ* during the curing process, and following curing.

4.1 Uncured paste

Measurements on the uncured paste generally relate to the rheological properties of the paste. The most common of these measurements is a slump test, which is defined by ASTM C143, and is useful for determining the workability of the material during its application. This test, with some slight modifications such as using a calibrated video camera to record height change, should be readily automatable, though automating the consolidation of the material for this test would be challenging. Work has also been done to describe a miniature slump test that would also be readily automatable.[21]

4.2 Curing cement

Tests on the cement *in situ* during curing typically involve more advanced instrumentation but are generally easily automated. Vicat measurements are a simple and common measurement method for measuring setting times of materials, and are described by ASTM C191. This method involves using needles to repeatedly probe the cement paste until it no longer penetrates (initial setting time) or marks (final setting time) the surface of a cement sample in order to measure the setting time. Similarly, this method can be used to determine if a cement paste is inadequately hydrated. There are commercially available automated tools for performing this test once the cement has been dispensed.

Another set of tests that could be useful to perform during the curing process is ultrasonic pulse velocity (UPV) measurements and calorimetry. UPV provides information on strengthening of the materials during curing.[22] These tests are easily parallelizable with enough pulse generators and transducers and systems are commercially available. Similarly, calorimetry can provide useful kinetic information about curing, as described in ASTM C1702 and C1679. While neither of these methods provide critical information for matching mixtures with applications, this information can be valuable to help understand the scientific underpinnings of the composition–properties relationship. The downside is that these methods require relatively costly instrumentation that remains dedicated to each sample for the duration of the measurement.

4.3 Cured product

There are a great many tests that can be performed on the cured material. We note here that the properties of the cured material can vary over time, and is frequently tested on the timescale months, though early indications of properties should be apparent much earlier.

Strength of the repair material is likely to be the most important characteristic. There are a variety of tests for these materials including in compression (ASTM C39 and ASTM C109) and flexion (ASTM C348). These tests are both relatively straightforward to automate and require little preparation. A sample handling robot could also be designed to load the various testers though this may be an example of a place where, given a limited budget, human intervention could be readily used. A slower but automatable and high-fidelity measurement of cement strength would be mapping of cement hardness, likely of a cross-section using Vickers or nano-indentation. This will provide important information about the distribution of mechanical properties within the material.

For repair applications there are several properties that are important. First, the shrinkage or expansion associated with the curing process must be minimized to avoid inducing stresses, either internal or external to the patch.[14] Autogenous strain provides a measure of the change in length of a sealed specimen (ASTM 1698). The apparatus for this measurement is simple and can reasonably cheaply be duplicated. Samples can be electronically monitored to record change in length as a function of curing time. In addition to these volumetric changes during curing, patch material must be matched to the host material based on coefficient of thermal expansion to minimize stresses during temperature changes, both those occurring during curing as well as weather. Last, adhesion of the repair material to the existing material is important. This can be tested through adhesion pull-off tests, reviewed in ref. 23. Generally these measurements are carried out by depositing a bolus of material onto a substrate with a defined interface area, then once it is cured measuring the force to separate them.

While in principle, modeling can be used to directly predict properties from mixture composition, the ML model can be made much more powerful by including structural information, as described below. X-ray diffraction (XRD) is a method of obtaining crystallographic information, including phase fraction, for the crystalline phases in the material. XRD data collection is outlined in ASTM C1365 though automating analysis using Rietveld refinement is still an open area of research.[24] Quantitative X-ray fluorescence (XRF) mapping can also provide important complimentary information about the elemental make-up of the sample.[25] Overall composition can be measured but application of a micro-focused source enables individual grains to be probed and correlated with the XRD analysis.

5 Analysis, modeling, and design of experiments

Once data has been collected experimentally, the first task is to analyze and reduce the data. While some measurement methods, such as compression testing, will natively provide simple scalar measurands, others, such as X-ray diffraction measurements will provide complex data. For the purpose of ML modeling and other downstream tasks, it is advantageous to reduce this data through quantitative analysis before ingestion

into such models. Some of these approaches to data reduction have been solved, such as for XRF,[25] while others are ongoing research areas, such as XRD.[24] Ensuring accuracy in these novel data analysis algorithms provides a challenge to successful implementation.

Once the data have been sufficiently reduced, ML methods can ingest the data to train models. Here, we plan to create a surrogate model that can correlate the input materials and processing with the properties and performance of the material. These types of models are an ongoing research effort, both within the cementitious materials community[16] as well as the broader materials science community. Specifically we envision creating a ML based model that can take into account the specific chemistry and structure of the input materials to enable design with any future material. This ML agent will likely composed of Gaussian process models, which are particularly well suited identifying unexplored regions of parameter space and have been successfully implemented in several autonomous material science systems.[26,27] More tailored models can be added to boost the predictive power of the model in this high-dimensional synthesis space: Models such as CAMEO[28] and the scientific value agent[29] operate in chemical composition space and account for step changes in properties across phase boundaries, while models like ALIGNN[30] explicitly model the atomistic structure to make property predictions. To match the complexity of the design space and the multimodality of the data streams, we anticipate that a hierarchical model would be most appropriate. These ML models could be organized into such an overarching hierarchical model using the MULTITASK framework.[31] These models will learn the relationships between the mixture input parameters and the cementitious materials performance through structural and thermokinetic properties of the paste and cured product. This approach also more easily allows for integration of physiochemical laws into the ML algorithms[24] at each level which further increase the predictive power of the models as well as increase knowledge generation through interpretability.

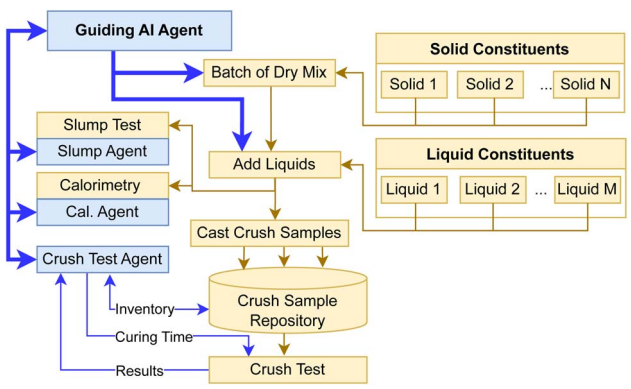

Fig. 2 A representative schematic showing the connection between different stages of the physical platform and the ML with three representative measurement modalities. Blue lines represent data flow brown lines represent physical material flow. The blue agents are responsible for measurement control, sample tracking, and data analysis.

As an autonomous system, the ML agent will also be responsible for directing and coordinating the experimental actions of the platform, as illustrated in Fig. 2. As mentioned above, the ML agent, using the hierarchical MULTITASK framework, will be responsible for selecting the combination of material to be tested and the battery of characterization tests that will be performed on that material. It will do this with an aim to identify candidate materials that will not just have the highest performance but also those that can provide the highest information content to the model. One challenge will be managing the utilization of long duration and hardware limited tests (*e.g.*, calorimetry). Part of this process for the ML will be looking for patterns to determine which low latency tests can be used to rapidly acquire complementary data, reserving the more equipment and time intensive tests for the most important samples.

6 Conclusion

Development of repair materials is both critically necessary – in terms of human, environmental, and fiscal costs – and a dynamic design challenge. Repair materials must be matched with their particular application over a range of properties. An autonomous system for synthesis and testing of cementitious materials, paired with a surrogate ML model, would be a powerful tool for rapidly formulating fit-for-purpose repair material. The needed properties can be fed into the trained model through a inverse design process to identify regions of formulation space that match the specific needs of a given job. The robotic platform can than be tasked with validating and optimizing the specific formulation.

The cost of building any autonomous system can vary greatly depending on the amount of custom hardware, who will be doing the engineering, construction, and coding work (*i.e.*, a commercial vendor *versus* graduate labor). We estimate the cost of the mixing plant, which is based on available components, not including design or construction labor to cost approximately $300k (USD) including the gantry. The cost of each station will vary dramatically based on its complexity. For instance a slump testing station or the compression testing station may cost as little as $20k but an station to process samples and measure XRD may be several hundred thousand dollars. That being said, if built modularly, these characterization stations can be added over time.

Ultimately, realization of such a platform still has several major challenges. As mentioned above, care will need to be taken in the design of the physical system to avoid cross contamination of samples and to ensure that the automated characterization stations are capable of accurately reproducing the results of standard measurements. At the end of the day, details of the platform will constrain the experimental search space, and as with all autonomous platforms, there is a risk optimal solutions will be outside of the accessible synthetic envelope. The ML necessary to control this system will also present a challenge. While this is an active area of work by a large community, these algorithms are on the cutting edge and will require additional research to customize them for this application.

Data availability

As this is a perspective article, no primary research results, data, software or code have been included.

Author contributions

HJ: writing – original Draft. All authors: conceptualization and writing – review and editing.

Conflicts of interest

The authors declare no competing interests. The opinions, recommendations, findings, and conclusions of this work do not necessarily reflect the views or policies of NIST or the United States Government.

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