ENHANCEMENT OF MECHANICAL PROPERTIES OF HIGH–VOLUME FLY ASH CONCRETE USING GLASS NANO POWDER AND EFFECTIVE MICROORGANISMS

ALI MOHAMMED AHMED SAGHIR ONAIZI

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> School of Civil Engineering Faculty of Engineering Universiti Teknologi Malaysia

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DEDICATION

I dedicate my thesis work to my late father MOHAMMED A. ONAIZI, whose lifelong priority was to see such an accomplishment realized. My dear father did not only raise and nurture me but also taxed himself dearly over the years for our education and academic development. It is also dedicated to my mother, for whom I cannot express how grateful I am. My beloved mother has been a source of motivation and strength, showing her motherly care and support in incredible ways during all moments. A special feeling of gratitude and appreciation to my role model, my uncle, Prof. SAGHEER A. ONAIZI, who deserves the most credit for constantly sponsoring, instructing, and following up on this work. Super thanks forever. It is also to my brothers AHMED, ABDUL-RAHMAN, and all my sisters.

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ABSTRACT

The low strength at early ages poses a major challenge to cement replacement with high volume fly ash of concrete (HVFA). The attempts to address strength reduction of HVFA concrete have not yet framed as radical solutions, considering the engineering solutions and the economic cost. So, this study aimed to investigate strategies to use local waste materials, which are more abundant and less expensive, to compensate for the strength loss associated with using HVFA as cement replacement. Both glass bottle waste nano-powder (GBWNP), and effective microorganisms (EMs) may offer a promising material for increasing strength at early ages due to their availability and minimal cost. In addition, GBWNP has sufficient pozzolanic properties to assist the pozzolanic reaction that can compensate for the early age strength reduction associated with HVFA concrete. To achieve the study goals, a series of modified concrete were prepared with 50% fly ash (FA), as cement replacement, 10% EMs as mixing water replacement, and 2%, 4%, 6%, 8%, and 10% of GBWNP as nano additives. The fresh properties of the new synthesized mixes were tested in terms of slump value and fresh-state density. The hardened properties examined are mechanical strength including compressive, flexural, and split strengths at 3, 7, 28, 56, and 90 days. The modulus of elasticity and water absorption were evaluated at 28 days. To generate more knowledge about the microstructures of the new modified specimens, various technical tests, including scanning electronic microscope (SEM), the energy dispersive X-ray spectra (EDS), X-ray diffraction (XRD) and thermogravimetric analysis (TGA) at ages of 7 and 28 days. Results of the examined specimens show that the integrated addition of GBWNP and EMs significantly improves strength indexes at all the tested ages. The specimens with 10% EMs as mixing water replacement and 4% GBWNP demonstrate mechanical performance comparable with that of the control samples at almost all curing ages, whilst the mix prepared with 6% GBWNP outperformed the control mix of normal concrete at all curing ages. The microstructural analysis showed that hydration products increase, and the microstructure compactness and homogeneity enhance by inclusion both GBWNP and EMs, especially in case of the inclusion of 10% EMs and 6% GBWNP. Furthermore, a decreasing trend of calcium to silicon ratio (Ca:Si) and calcium to aluminium ratio (Ca:Al) due to the inclusion of GBWNP while an increasing trend of silicon to aluminium ratio (Si:Al) was observed, which confirm the role of GBWNP to boost FA to react faster, hence, improving strength properties. By employing fly ash and glass bottle waste, this study also seeks to contribute to the improvement of the environment by encouraging the recycling of waste in concrete sectors, providing efficient solutions to the landfill problems.

ABSTRAK

Kekuatan yang rendah pada usia awal menimbulkan cabaran besar kepada penggantian simen dalam konkrit dengan kandungan tinggi abu terbang (HVFA). Percubaan untuk menangani pengurangan kekuatan konkrit HVFA belum lagi dirangka sebagai penyelesaian radikal, memandangkan penyelesaian kejuruteraan dan kos yang ekonomi. Jadi, kajian ini bertujuan untuk menyiasat strategi untuk menggunakan bahan buangan tempatan, yang lebih banyak dan lebih murah, untuk mengimbangi kehilangan kekuatan yang berkaitan dengan penggunaan HVFA sebagai pengganti simen. Kedua-dua serbuk nano sisa botol kaca (GBWNP), dan mikroorganisma berkesan (EMs) mungkin menawarkan bahan yang menjanjikan untuk meningkatkan kekuatan pada peringkat awal umur kerana ketersediaannya dan kos yang minimum. Di samping itu, GBWNP mempunyai sifat pozzolanik yang mencukupi untuk membantu tindak balas pozzolanik yang boleh mengimbangi pengurangan kekuatan pada usia awal yang dikaitkan dengan konkrit HVFA. Untuk mencapai matlamat kajian, satu siri konkrit yang diubah suai telah disediakan dengan 50% abu terbang (FA), sebagai pengganti simen, 10% EMs sebagai pengganti bancuhan air, dan 2%, 4%, 6%, 8%, dan 10% daripada GBWNP sebagai bahan tambah nano. Sifat segar campuran tersintesis baharu ini kemudian diuji dari segi nilai kericihan dan ketumpatan dalam keadaan segar. Sifat keras yang dikaji adalah dari segi kekuatan mekanikal termasuk kekuatan mampatan, lenturan, dan tegangan pada 3, 7, 28, 56, dan 90 hari. Modulus keanjalan dan penyerapan air telah dinilai pada 28 hari. Untuk menjana lebih banyak pengetahuan tentang mikrostruktur spesimen baharu yang diubah suai, pelbagai ujian teknikal, termasuk pengimbasan elektronik mikroskop (SEM), spektrum sinar-X penyebaran tenaga (EDS), pembelauan sinar-X (XRD) dan analisis termogravimetrik (TGA) pada umur 7 dan 28 hari. Keputusan daripada spesimen yang diperiksa menunjukkan bahawa penambahan bersepadu GBWNP dan EM meningkatkan indeks kekuatan dengan ketara pada semua peringkat umur yang diuji. Spesimen dengan 10% EMs sebagai pengganti air bancuhan dan 4% GBWNP menunjukkan prestasi mekanikal yang setanding dengan sampel kawalan pada hampir semua umur pengawetan, manakala campuran yang disediakan dengan 6% GBWNP mengatasi prestasi campuran kawalan biasa konkrit pada semua peringkat umur pengawetan. Analisis mikrostruktur menunjukkan bahawa produk penghidratan meningkat, kepadatan dan keseragaman mikrostruktur bertambah dengan kemasukan kedua-dua GBWNP dan EMs, terutamanya dalam kes kemasukan 10% EMs dan 6% GBWNP. Tambahan pula, corak penurunan nisbah kalsium kepada silikon (Ca:Si) dan nisbah kalsium kepada aluminium (Ca:Al) disebabkan oleh kemasukan GBWNP manakala corak peningkatan nisbah silikon kepada aluminium (Si:Al) telah diperhatikan, yang mengesahkan peranan GBWNP untuk menggalakkan FA untuk bertindak balas dengan lebih pantas, oleh itu, meningkatkan sifat kekuatan. Dengan menggunakan abu terbang dan sisa botol kaca, kajian ini juga bertujuan untuk menyumbang kepada penambahbaikan alam sekitar dengan menggalakkan kitaran semula sisa dalam sektor konkrit, menyediakan penyelesaian yang cekap kepada masalah tapak pelupusan.

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LIST OF ABBREVIATIONS

ASTM	_	American Standards for Testing of Materials	
BS EN	_	British Standard	
СН	_	Calcium Hydroxide	
С–А–Ѕ–Н	_	Calcium Aluminate Silicate Hydrates	
С-S-Н	_	Calcium Silicate Hydrate	
CNTs	_	Nanotubes	
CNFs	_	Nanofibers	
DTA	_	Differential Thermal Analysis	
EDS	_	Energy-Dispersive X-Ray Spectra	
EMs	_	Effective Microorganisms	
FA	_	Fly Ash	
HVFAC	_	High-Volume Fly Ash Concrete	
GBWNPs	_	Glass Bottle waste Nano-Particles	
MoE	_	Modulus of Elasticity	
OPC	_	Ordinary Portland cement	
TGA	_	Thermal Gravimetric Analysis	
SEM	_	Scanning Electron Microscopy	
XRD	_	X-ray diffraction	
XRF	_	X-ray Fluorescence	

LIST OF SYMBOLS

Ws	-	Dry-surface Weight
Wd	-	Oven-dried weight
ρ	-	Density
m	-	Mass of specimen
v	-	Volume of specimen
L	-	Length of specimen
Н	-	Height of specimens
fc	-	Compressive strength
Р	-	Applied Load
А	-	Area of specimen
fct	-	Splitting tensile strength
f _{cf}	-	Flexural strength

CHAPTER 1

INTRODUCTION

1.1 Introduction and background of the problem

Globally, diverse concretes are still the major manufactured materials that are predominantly consumed worldwide other than water [1, 2]. Cement is a vital component in concrete production, but in recent decades many challenges facing concrete completely based on Ordinary Portland Cement (OPC) have begun to appear. As a result of the increase in the complexities of construction environments, durability became represents a concern for conventional concrete. Besides that, the environmental concerns related to cement production increase continuously, especially in terms of carbon dioxide (CO₂) footprint. In accordance with the 2015 Paris Agreement, global carbon emissions from the cement industry must be reduced by at least 16% by 2030 if Earth is to remain within 1.5 °C or 2 °C of pre-global warming [3]. However, with continuous urban land expansion, population growth and economic development, statistics indicate that global cement production will increase from the current 4.08 billion tonnes to 5 billion tonnes in the next 30 years [4]. Furthermore, in accordance with the Global Commission on the Economy and Environment, if developing countries increase their infrastructure to the current global average levels, then the construction industry will emit 470 gigatonnes of CO₂ into the atmosphere by 2050. Apart from producing massive amounts of greenhouse gases (e.g. CO₂, SO_x and NO_x emissions) and consuming high amounts of energy during the heating process, cement contributes to rapid landscape degradation, dust production during transportation, noise generation in quarries and raw material exhaustion [5]. All these conditions are major environmental issues that exert an impact on ecosystems that is critical to human well-being [6].

Conventional concrete demonstrates poor performance in hostile environments, particularly in the case of marine, coastal, and acid rain-exposed structures. Dams, concrete sidewalks, bridge posts, underground concrete foundations, and highway tunnels, especially those submerged in soil or seawater or near coasts or manufacturers that release chemical oxides, have deteriorated dramatically due to acid and sulphate attacks in recent decades. Cement-based concrete is sensitive to chemical attacks because of the calcium compounds present. In addition, the high porosity and crack ratio can encourage water and chemical ions to infiltrate into the matrix of the concrete, which can result in damage to the components of the concrete as well as corrosion of the reinforcing steel bars. In the case of the freeze-thaw cycle action, water trapped inside concrete matrix induces internal stress inside the matrix, leading to the development of further cracks. Furthermore, the enhanced microstructural instability and rapid disintegration that arise from calcium compounds dissolving rapidly in the acidic environment further complicates the situation. As a result, the chemical attack raises worries about the durability of cement-based concrete, which places a threat on a significant number of structures that are composed of concrete. As a result, the development of supplementary cementitious material-based concrete is one of the radical solutions to meet the challenges of durability.

Using a low percentage (<30%) or a high percentage (>30%) of fly ash (FA) in concrete is a pioneering move that has already approach to develop high durable concrete [7]. Even though FA has been the subject of extensive research over the past few decades, experts have found some contradictory findings regarding the mechanical and durability properties of concrete. Furthermore, the characteristics of FA vary based on the source. It is accepted that FA qualifies as an supplementary cementitious materials (SCM) and can be substituted for cement in the production of cementitious materials[8]. Several studies revealed FA enhances workability, reduces hydration heat and thermal cracking in cementitious materials at early ages, and improves the mechanical and durability properties of cementitious composites primarily at later ages[9, 10]. FA also decreases bleeding, which makes concrete more workable[11]. During the hydration process, the binder causes the concrete to shrink, where high shrinkage can lead to significant cracks in the structure of the concrete. So, the slow pozzolanic nature of FA is advantageous for shrinkage reduction[12]. Using high volumes of FA in concrete, specifically around 50% as cement replacement, reduced shrinkage by 30% [13]. On another aspect, Calcium hydroxide (CH) is considered a compound that has a high propensity to react with aggressive ions, which leads to the rapid deterioration of concrete due to a series of undesirable reactions. However, by converting CH to cementitious gels, through reacting with silica and aluminum present in FA, leads to fill pores and refine cracks, thus enhance concrete durability. Beside consumption of CH, filling pores and cracks, concrete resistance to aggressive ions penetration enhances, thus concrete durability increases [14]. However, since FA content's pozzolanic reaction is slower than OPC, high–volume fly ash (HVFA) concrete exhibits a marked decline in strength properties at early ages[7]. Even though the strength decreases at early ages, the strength gain is maintained for a significantly longer period of time compared to normal concrete [15]. That means FA requires an extended curing period to hydrate, which causes delays in the pursuance of construction applications. Overcoming the decline in strength at early ages is still a challenge, and no precise solutions have yet been framed with clear standards and limitations.

Several studies have provided various solutions to overcome the reduction in strength of HVFA at early ages. Some studies have suggested increasing the fineness of FA particles to increase its surface area, accelerating the pozzolanic reaction and forcing the fine FA particles to act as seeds and fillers [16-18]. Other studies have proposed increasing the alkalinity of the concrete mixture by adding extra lime (CH) to catalyse FA particles and make them react faster [19]. Amongst all these methods, nanomaterials have sparked the most interest due to their remarkable capability to fix concrete flaws at early ages, particularly those caused by FA. A wide range of nanoadditives, such as nano-SiO₂, Al₂O₃, Fe₂O₃, Fe₃O₄, ZnO₂, ZrO₂, Cu₂O₃, CuO, CaCO₃, SF and CTs [20-25], have been harnessed due to their remarkable effects on improving the strength properties and durability of nano-modified concrete. Recently, the trend is to using waste materials sush as waste ceramic and glass, which have pozzolanic properties, as nanomaterials to overcome the flaws of concrete including HVFA concrete. The presence of noncrystalline aluminium and silicon (with high amorphous silica content) in pulverised glass waste from bottles makes such waste an effective pozzolanic material [26, 27]. Research on using glass bottle waste as nanomaterial additive to improve the early strength properties of cement is still insufficient. However, the studies that conducted on using glass bottle waste nano powder (GBWNP), including Huseien et al. study [28] revealed that adding glass bottle waste nano-powder exerts a positive effect on compressive, flexural and splitting tensile strengths, modulus of elasticity (MOE) and water absorption (WA). In accordance with the aforementioned studies [27-30], the improvement in strength and microstructure properties may be attributed to the pozzolanic reaction process, the production of extra C–S–H gel and the physical filling effect of pores and cracks, which reflect positively on the strength properties, homogeneity and compactness of cementitious systems.

In the last decade, the concept of self-healing concrete based on effective microorganisms (EMs) have developed. Effective microorganisms have shown a significant ability to improve concrete durability and restore the strength of damaged concrete. Besides, the EMs were also exhibited an ability to improve the strength properties even if undamaged concrete [31, 32]. Several researchers reported that incorporating 3%–15% EMs into the concrete matrix enhanced mechanical properties and microstructure at early ages [31, 33-35]. The enhancement of strength properties and microstructure at early ages was hypothesised to be due to calcite precipitation by EMs inside the matrix, increasing compressive [31, 36], splitting tensile and flexural strengths [36-38]. However, most studies that indicated the effect of microorganisms on mechanical strength were conducted on cement pastes, mortars or low-volume FA concrete. Besides, there is lack in understanding the mechanism of the EMs role in improving the strength performance of undamaged concrete. Furthermore, the investigation of the efficacy of effective microorganisms in improving the strength properties at early ages still not exist.

1.2 Problem statement

Fly ash (FA) is considered a pozzolanic material. It contains high content of silica and aluminium content that can react chemically with calcium hydroxide (CH) in presence of water to produce extra cementitious gels. However, the reactivity of silica and aluminium content embedded in FA is lower than cement particles, which means that the pozzolanic reaction of FA particles takes long time to react more than that in cement. Furthermore, the activation FA content depends on the concentration of calcium hydroxide that raise the alkalinity of concrete, thus forcing FA particle to

react faster. However, when the replacement level of FA increases the calcium content decreases, thus, the CH formed by cement hydration became insufficient for activating all the FA contents to trigger pozzolanic reaction [39, 40]. Consequently, many unreacted FA particles remained in the paste, causing weaker bonds between aggregates, increasing the pore size and total porosity of the matrix, reducing mechanical strengths in consequence [19]. However, with time progress, more CH content precipitated around the unreacted FA particles, and the pozzolanic process continuous, thus the unreacted FA particles react [41]. But the long time required to reach the targeted compressive strength hinders progress in the implementation of the construction applications. Therefore, the search for cost-effective solutions to overcome the reduction associated with replacing OPC with high-volume fly ash (HVFA) has become an urgent need.

Several previous studies addressed various solutions to overcome the reduction in strength in early ages of concrete containing a high-volume fly ash such as increase the fineness of FA particles [16-18] and increasing the alkalinity of the concrete mixture by adding extra lime (CH) [19]. Among all of these methods, nanomaterials have sparked the most interest due to their remarkable ability to fix concrete flaws at early stage, particularly those caused by fly ash. However, most of the nanomaterials that have been introduced are rare or expansive to prepare. So, the utilize of local waste such as GBWNP and EMs seems to be more effective solution. Studies focusing to use GBWNP and EMs to improve the early strength properties of HVFA concrete is still not sufficient. Besides, the previous studies had examined the effect of the EMs and nanomaterials on mechanical performance of cement-based concrete separately. Thus, this research aims to improve the mechanical and microstructure properties of HVFA concrete by the integration of both glass bottle waste nano-powder and effective microorganisms.

1.3 Research Objectives

Based on the abovementioned problem statement the following objectives are set:

- (a) To determine the optimum percentage of GBWNP that achieved better strength performance of HVFA concrete at early ages.
- (b) To overcome the reduction in mechanical performance resulting due to replacing cement with 50% of FA by using GBWNP and EMs.
- (c) To investigate the role of both GBWNP and EMs in enhancing the microstructure properties of HVFA concrete.

1.4 Research Scope and Limitation

This research (experimental) examined the influence of co-adding both glass bottle waste nano powder and effective microorganisms on the possibility of developing a new modified concrete with enhanced mechanical strength and durability. This novel modified concrete were made by combining OPC, FA, and GBWNP as binders with a mixture of tap water and EMs broth as a mixing solution. The high-volume fly ash modified concrete has been synthesized by replacing 50% of OPC with FA, that has been collected from the Tanjung Bin power station in Johor (Malaysia). Based on a literature review, fly ash as cement replacement levels of more than 50% leads to lack of calcium content, thus not guarantee achieving the target strength. Therefore, FA content has been fixed at the level of 50%. The water-cement ratio also was fixed at 0.50 to achieve a comparison purpose between the different synthesized mixes and avoid the side effects of change of w/c ratio. Similarly, based on a literature review, effective replacement levels of EMs have been indicated in the range of 5-15% of the mixed water content, so the content of the EM solution has been kept at 10 % wt. of water mixing. Then, various percentages of glass bottle waste (2, 4, 6, 8 and 10% wt. of binder) have been added to achieve the target strength between 30-40 MPa at 28 days.

The physical, chemical, and mineralogical characteristics of raw materials were determined in order to get a better understanding of their properties. The effects of co-addition of glass bottle waste nano-powder (2, 4, 6, 8 and 10% wt. of binder) and EMs on workability, mechanical properties and microstructures of synthesized

modified concrete were examined, and the optimum percentage of glass bottle waste nano-powder has been determined. The proportions of the mix were calculated using the method described in Marsh (2007), also known as the Department of Environment (DOE) method. The control mix were then calibrated to achieve a compressive strength higher than 30 MPa after 28 days with a slump between 30–60 mm.

The workability of concrete was evaluated in terms of slump to provide an assessment of the ease of handling of concrete in its fresh state in accordance with ASTM C143 [42]. Compressive strength test was conducted in accordance with ASTM C109 specifications [43], and due to the compressive strength is the most critical factor in concrete, the optimal percentage of GBWNP was determined on the basis of compressive strength results. Depending on these results, the flexural strength, splitting tensile strength, modulus of elasticity (MOE) and WA tests and microstructural tests were preceded with the optimal percentage of GBWNP. Flexural strength was tested in accordance with ASTM C78 [44]. Tensile strength was evaluated in accordance with ASTM C496 [45]. MOE was examined in accordance with ASTM C469/C469M [46]. The compressive, splitting tensile, flexural strength were determined at 3, 7, 28, 56 and 90 days. The modulus of elasticity, hardened density and water absorption were determined at 28 days. Thermal Gravimetric Analysis (TGA), X-ray Diffraction (XRD), and scanning electron microscopy (SEM) measurements were used to evaluate the microstructures of the designed mixes at 7 and 28 days. In evaluating the available literature, the International Union of Laboratories and Experts in Construction Materials, Systems, and Structures (RILEM) was used. Furthermore, the findings of the study have been compared to similar research to provide further evidence of their precision.

1.5 Significance of study

As abovementioned, this research intends to generate new information on the use of glass bottle waste nano powder, which is considered widely available, inexpensive, and provides environmental solutions to reduce solid waste. Furthermore, it will contribute to the advancement of standard requirements for multi-blend modified concrete through systematic techniques of sample preparation from waste materials, suitable and meticulous materials characterizations, and subsequent data analysis. This understanding is likely to spur the development of environmentally friendly and low-cost modified concrete for a wide range of applications in the construction industry. This would be extremely beneficial to Malaysia's long-term growth, as waste disposal issues related to landfilling would be avoided and minimised. The novel findings of this study are expected to provide a foundation for future research and improved understanding of the behaviour of a multi-blend modified concrete made from waste in a cost-effective and environmentally friendly manner.

1.6 Thesis Organization

Chapter 1 provided introduction, background of the problem, problem statement, study's objectives, scope and limits, and significance of the study.

Chapter 2 conducted a thorough analysis of the available relevant literature and covered the features of modified concrete and pozzolanic materials. It also summarised prior researches on FA, EMs, and nanomaterials-based modified concrete. This chapter was also outlined the study's gaps.

Chapter 3 highlighted a thorough discussion of the materials and sample synthesis procedures, as well as the tests utilised to characterise the samples. The basic principles of several tests were emphasised, which are important for evaluating the performance of modified concrete. Furthermore, characterized the physical, mineral and chemical properties of OPC, FA, EMs, and GNP in details. The characterization of coarse and fine aggregate also provided in this chapter.

Chapter 4 presented the significant experimental outcomes and discussions on the fresh and hardened properties of modified concrete. The results of workability/slump, the compressive strength, splitting tensile, flexural strength, modulus elasticity and water absorption were presented in discussed. Moreover, the microstructures of the

new designed concretes were revealed using SEM images, EDS analysis, TGA/DTA, and XRD spectra, and describing the percentage and type of hydration products for each mixture.

Chapter 5 concluded the thesis and provided some recommendations for further research in modified concrete using waste materials.

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