PERFORMANCE AND MODELING OF VOLCANIC ASH AND LIMESTONE POWDER WASTE BINARY BLENDED ALKALI ACTIVATED MORTAR

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DEDICATION

To Almighty Allah who teaches man what he knows not To Prophet Muhammad who is a blessing to the universe To my parent who trained and nourished me To my lovely wife for her support and patient To my friends who are always there for me To all my teachers who believe in me To all lives lost to COVID – 19.

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ABSTRACT

The greenhouse gas emissions and energy consumption from the production of ordinary Portland cement (OPC) have caused the need to have an alternative sustainable binder system that is eco-friendlier. While alkali-activated material (AAM) is rapidly emerging as a potential eco-efficient and economically viable alternative material to OPC, getting the raw materials for AAM from local sources has been a significant challenge. Local sourcing of raw materials from industrial by-products ensures the sustainability of AAM. Furthermore, to enhance the commercialization of AAM, the mechanical and the durability performance of the AAM synthesized from the local materials need to be established. This research seeks to advance state of the art toward using locally sourced waste materials to develop alkali-activated mortar. Two of the identified viable solid wastes locally available in Saudi Arabia are volcanic ash (VA) and limestone powder (LSP). This study aimed to synthesize alkali-activated mortar using VA and LSP and to develop models using artificial intelligence and statistical techniques that can quickly and accurately predict the compressive strength of alkaliactivated volcanic ash and limestone powder. Firstly, experimental work was carried out to synthesize and evaluate the developed mortar's fresh, mechanical, and durability performance. The impact of constituent variables such as binder ratio varied from (0 to 1), sodium hydroxide concentration [4-14]M], silica modulus (0.52 - 1.18), curing temperatures (ambient room temperature and oven cured temperature maintained from 45 to 90 °C at an interval of 15 °C for 24 hrs), Fine aggregate -tobinder ratio (1.4 - 2.2) and alkaline activator to binder ratio (0.45 - 0.55) on the compressive strengths and microstructure of the developed mortar was investigated. The synthesized alkali-activated VA and LSP binders were examined critically to study the impact of the constituent variables on the product formed, bond characteristics, and microstructures using x-ray diffractometer (XRD), scanning electron microscope coupled with energy-dispersive x-ray spectroscopy (SEM+EDX), and Fourier transforms infrared (FTIR) spectroscopy. Secondly, different models capable of estimating one-day, three-day, 14-day, and 28-day compressive strength (CS) were developed using a hybrid genetic algorithm (GA), support vector regression (SVR) algorithm, and stepwise regression algorithm. The optimization of the mix parameters gave the maximum 90-day compressive strength of 31.3 MPa with 60% LSP and 40% VA using 10 M NaOH_(aq), silica modulus of 0.89 and cured at 75 °C for 24 hrs duration. Besides, about 77% of 28-days compressive strength (27 MPa) could be achieved in 24 hrs using heat curing. Samples synthesized with sole 10 M NaOH(aq) resulted in a binder with a low 28-day compressive strength (15 MPa) compared to combined usage of Na₂SiO_{3(aq)}/10 M NaOH_(aq) activators. Curing at a low temperature (25 °C to 45 °C) does not favour strength development, whereas higher curing temperature enhances strength development. The findings also revealed that the synergistic effect of VA with LSP emanated from silica and alumina required to form an aluminosilicate framework, which required cation sourced from LSP (Ca^{2+}) for charge balancing in the formed skeletal framework. The binder products formed are anorthite (CaAl₂Si₂O₈) and gehlenite (Ca0.Al₂O₃.SiO₂). Microstructural analysis revealed that the rough texture of activated VA initially characterized with high porosity turned to be filled up by the presence of LSP, thereby improving the microstructural density. SEM+EDX indicated that strong alkali (10 M NaOH) enhanced microstructural density compared to that of mild alkali (4 M NaOH(aq)). The binder synthesized with 60% VA and 40% LSP exhibited the highest resistance to sulfate and acidic attack. The developed hybrid GA-SVR models can estimate the compressive strength of mortar for one day, three days, and fourteen days, up to 96.64%, 90.84, and 93.40% degree of accuracy as measured based on the correlation coefficient between the measured and estimated value. The developed cubic with interactions stepwise regression model (V) was characterized with a high correlation coefficient of 97.2% compared to the other four models (I-IV). The developed mortar can be used where repair work is required due to its early strength. The outcomes of this study also contributed to waste valorization, dumpsite land reclamation, low CO₂ footprint, energy consumption reduction, reduction in environmental pollution, and the addition of more sustainable alternative binders for structural purposes. Furthermore, the outcomes of this work would provide a quick and efficient way of predicting the compressive strength of environmental friendly binders with minimal experimental stress and errors inherent in the laboratory.

ABSTRAK

Pelepasan gas rumah hijau dan penggunaan tenaga dari proses pembuatan simen portland biasa (OPC) mememerlukan penggantian alternatif kepada sistem pengikat yang lebih mesra alam. Bahan pengaktifan alkali (AAM) mula dikenali sebagai bahan berpotensi yang mesra alam dan ekonomi menggantikan OPC, walaupun terdapat cabaran untuk memperolehi bahan tersebut dari sumber tempatan. Bagi memastikan kelestarian AAM, bahan tersebut hendaklah diperolehi dari bahan pemprosesan industri tempatan. Tambahan pula, jika hendak dikomesilkan bahan AAM tersebut, kajian tentang ciri-ciri mekanikal dan ketahanlasakan sintesis dari bahan AAM mestilah diiktiraf umum. Oleh yang demikian kajian lanjut diperlukan untuk menghasilkan bahan pengaktifan alkali 'mortar'yang moden dari sumber tempatan. Terdapat dua jenis bahan sintesis dari Arab Saudi iaitu abu gunung berapi (VA) dan abu batu kapur (LSP). Matlamat kajian ini adalah untuk mensintesis pengaktifan alkali 'mortar' dari bahan VA dan LSP tersebut dan membina satu model menggunakan kaedah kepintaran buatan dan teknik statistik di mana ianya membolehkan kekuatan mampatan pengaktifan alkali tersebut ditentukan. Bahagian pertama ialah melakukan kajian untuk mensintesis, menilai ciri-ciri mekanikal dan ketahanlasakan prestasi 'mortar'. Kesan pembolehubah seperti nisbah pengikat dari 0-1, kepekatan sodium hidroksida (4-14 M), modulus silika (0.52-1.18), pengawetan suhu (suhu bilik dan suhu oven, 45-90 °C pada sela 15 °C selama 24 jam), aggregat halus kepada nisbah pengikat (1.4-2.2) dan pengaktif alkali kepada nisbah pengikat (0.45-0.55) terhadap kekuatan mampatan dan mikrostruktur 'mortar' adalah dikaji. Sintesis pengaktifan alkali VA dan LSP ini dikaji secara kritis dari segi impak pembolehubah terhadap produk tersebut, ciri-ciri ikatan dan mikrostruktur menggunakan alat saintifik XRD, SEM+EDS dan FTIR. Bahagian kedua, beberapa model dibuat bagi tempoh pengawetan iaitu satu hari, tiga hari, empat belas hari dan dua puluh lapan hari untuk diukur kekuatan mampatan menggunakan kaedah kacukan genetik algoritma (GA), vektor dan algoritma regresi. Campuran optimum berdasarkan parameter menunjukkan prestasi kekuatan mampatan yang tinggi iaitu 31.3 MPa ialah campuran 60% LSP dan 40% VA dengan 10M NaOH, silika modulus 0.89 pada suhu pengawetan 75 °C selama 24 jam. Selain itu, 77% dari nilai kekuatan mampatan 27 MPa pada tempoh 28 hari pengawetan pemanasan suhu boleh dicapai dalam masa 24 jam. Sampel yang disintesis dengan 10 M NaOH dalam masa 28 hari, memberi nilai kekuatan mampatan yang lemah iaitu 15 MPa berbanding kombinasi Na₂SiO₃/10 M NaOH. Pengawetan pada suhu rendah di antara 25 °C - 45 °C tidak membantu di dalam peningkatan kekuatan mampatan berbanding pengawetan pada suhu tinggi. Keputusan kajian keberkesanan gabungan bahan VA dengan LSP menunjukkan kandungan Silika dan Alumina diperlukan bagi membentuk alumina-silika struktur iaitu kation LSP (Ca^{2+}) untuk keseimbangan struktur tersebut. Hasil produk yang terbentuk dari pengikatan bahan tersebut ialah Anorthite (CaAl₂Si₂O₈) and gehlenite (CaO.Al₂O₃.SiO₂). Analisis mikrostruktur mendapati bahawa ciri-ciri VA yang mempunyai kelompangan yang tinggi telah menjadi lebih padat mikrostrukturnya apabila digabungkan dengan LSP. SEM+EDX menunjukkan bahawa kemolaran alkali (10M NaOH) boleh meningkatkan ketumpatan mikrostruktur berbanding kemolaran sederhana (4M NaOH). Pengikat yang disintesis oleh 60% VA dan 40% LSP memberikan nilai rintangan tinggi terhadap serangan sulfat dan asid. Model hybrid GA-SVR boleh tentukan kekuatan mampatan 'mortar' bagi tempoh pengawetan selama satu hari, tiga hari dan empat belas hari dengan ketepatan 96.64%, 90.84% dan 93.40% darjah ketepatan berdasarkan ukuran pekali kolerasi di antara pengukuran dan nilai anggaran. Binaan kiub dengan interaksi model aturan regrasi telah dicirikan dengan pekali kolerasi tinggi iaitu 97.2% dibandingkan dengan empat model lain (I-IV). Model 'mortar' vang dihasilakan tersebut boleh digunakan untuk kerja-kerja pembaikan kerana kekuatan mampatan pada awalnya menunjukkan peningkatan yang tinggi. Hasil kajian ini juga dapat menyumbang kepada penyelesaian masalah pembuangan sisa, penambakan sisa, pengurangan pengeluaran CO₂, pembaziran tenaga dikurangkan, pencemaran alam sekitar dapat dikekang. Di samping itu bahan buangan tersebut boleh digunakan semula untuk binaan struktur. Selanjutnya kajian ini juga boleh digunakan untuk mengurangkan tekanan kerja bagi menentukan kekuatan mampatan mortar dengan cekap, pantas dan mengurangkan ketidakpastian dan kesalahan di dalam ujian makmal.

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LIST OF ABBREVIATIONS

AAM	-	Alkaline activated materials
AAB	-	Alkaline activated binder
AAC	-	Alkaline activated concrete
VA	-	Volcanic ash
LSP	-	Limestone powder
AALN	-	Alkaline activated limestone and volcanic ash
AAVLM	-	Alkaline activated volcanic ash and limestone mortar
AAP	-	Alkaline activated paste
BFS	-	Blast furnace slag
CAA	-	Combined alkaline activator
CAS	-	Calcium Alumino-silicate phase
CMS	-	Calcium Magnesium silicate phase
C-S-H	-	Calcium-Silicate-Hydrate
C-A-S-H	-	Calcium-Alumino-Silicate-Hydrate
EDX	-	Energy Dispersive X-ray Spectroscopy
FA	-	Fine aggregate
FA	-	Fly-ash
NP	-	Natural pozzolan = VA
FA_L	-	Low carbon fly-ash (Class F)
FA_H	-	High calcium fly-ash (Class C)
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
FWC	-	Free or additional water content
GSS	-	Ground Steel Slag
M_s	-	Silica modulus
MM	-	Elemental molar-mass
OPC	-	Ordinary Portland Cement
POFA	-	Palm oil fuel ash
PSD	-	Particle Size Distribution
RHA	-	Rice husk ash

SEM	-	Scanning Electron Microscopy
SCM	-	Supplementary cementitious materials
SF	-	Silica fume
SSD	-	Saturated surface dry
SA	-	Surface area
ТА	-	Total aggregates
XRF	-	X-ray fluorescence
XRD	-	X-ray Diffraction
w/b	-	Water to Binder Ratio

LIST OF SYMBOLS

А	- Cross section area of specimen
Al	– Alumina
Al_2O_3	– Aluminium oxide
В	– Binder
B:A	– Binder to aggregate ratio
Ca	– Calcium
Ca(OH) ₂	 Calcium hydroxide
CaO	– Calcium oxide
CaO:SiO ₂	- Calcium to silicate ratio
C-A-S-H	- Calcium aluminium silicate hydrate
CO_2	– Carbon dioxide
C-S-H	- Calcium silicate hydrate
fb	– Bond strength
fc	- Compressive strength
Fe	– Iron
Fs	– Flexural strength
Ft	 Tensile strength
H_2SO_4	– Sulphuric acid
Hrs	– Hours
КОН	– Potassium hydroxide
MgSO ₄	– Magnesium sulfate
MPa	– Mega pascal
NaOH	– Sodium hydroxide
Na ₂ O	– Sodium oxide
Na_2SO_3	– Sodium silicate
SVR	- support vector regression
GA	– Genetic Algorithm

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CHAPTER 1

INTRODUCTION

1.1 General Introduction and Background of the Study

Concrete is the backbone of the built environment, especially in urban areas. The construction of infrastructures such as bridges, roads, dams, tunnels, high-rise buildings, airports, seaports, power plants, seawalls, wastewater plants, freshwater plants, and dykes for social and economic benefits consumed roughly 35 billion tons of concrete inclusive of other building materials such as steel, wood, and aluminum (Van Damme, 2018). This is generally due to its favourable compressive strength, durability, versatility, global availability, high fire resistance, and relatively low cost (Imbabi, Carrigan, and McKenna, 2012). Ordinary Portland cement (OPC) plays a vital role in the production of concrete. OPC binds the fine and coarse aggregate together in the presence of water through the hydration process. OPC world production was estimated to be 4.6 billion tonnes in the year 2015 with a projection of a four-fold increase by 2050 (CEMBUREAU, 2015). Despite the vital role played by OPC in infrastructural development, there are some setbacks associated with its production process. The major disadvantage of OPC is that the production process of OPC is energy-intensive and significantly leads to the emission of 5-8% of global CO2 into the atmosphere which poses a danger to the world ecological systems (Duxson et al., 2007; Damtoft et al., 2008; Andrew, 2017). The proliferation of CO₂ that accompanies high demand for cement from the emerging countries, especially China and the developing nations has called for alternative materials for construction and efficient use of energy (Duxson and Provis, 2008; Van Damme, 2018). To mitigate this environmental hazard that goes with greenhouse gas, the cement industries are recently focusing on the development of sustainable alternative binders such as alkali-activated materials (AAM)) due to its ability to use industrial waste materials as based materials (Rafiei, 2016).

The inclusion of supplementary cementitious materials (SCM) such as palm oil fuel ash (POFA), silica fume, slag, fly-ash, and metakaolin as partial substitution has reduced the global cement consumption and enhanced the structural and structural performance of concrete. The usage of the SCM is largely dependent on the availability of the materials in the country. For example, fly ash's annual output in China is estimated to be 600 million tons Ma et al. (2017), India, the USA, and Malaysia annually produced 112 million tons, 75 million tons, and 6.8 million tons respectively (Ghazali, Muthusamy and Wan Ahmad, 2019). The use of fly ash up to 30 to 70% substitution improved the workability of fresh concrete, enhanced mechanical strength, reduce the heat of hydration, and enhanced the durability performance of hardened mortar concrete (Halstead, 1986). Alkali-activated material (AAM) has been identified as an eco-efficient and economically viable alternative for partial or full replacement OPC due to its excellent strength, thermal and low permeability (Provis and van Deventer, 2014; Luukkonen et al., 2018a). AAM is a system formed by the reaction of soluble alkali activator and aluminosilicate-based materials (Provis and van Deventer, 2014). AAM is classified into low calcium (fly ash, metakaolin, and volcanic ash) and high calcium (blast furnace slag) binders. The main products in low binder AAM could be mainly potassium/sodium aluminosilicate hydrate with impregnation of alumina (N-A-S-H and K-A-S-H) within the formation. In high calcium binder such as blast furnace slag that is synthesized with a mild alkali, the main product is calcium alumina silicate hydrate (Najimi, Ghafoori and Sharbaf, 2018). Apart from the enhanced structural and durability performance of concrete developed using AAM, it is eco-efficient, economically viable, and reduced the proliferation of CO2 presents in the atmosphere.

Besides, the challenges of solid waste disposal generated from manufacturing and agro-allied industries have necessitated the need to look for a way on how to valorize them for construction purposes. These wastes include agricultural waste materials such as rice husk ash (RHA), palm oil fuel ash (POFA), or industrial waste such as silico-manganese slag (SiMn), ground granulated blast furnace slag (GGBFS), fly ash (FA), silica fume (SF), coal bottom ash, paper sludge ash and mine tails from mining activities, limestone powder waste (LSP) and volcanic ash (VA). These materials are of relevance to AAM synthesis owing to the aluminosilicate components that could make them potentially useful as a based materials for binder development. This is of utmost interest to civil engineers simply because of their pozzolanic nature to generate additional calcium-silicate-hydrate (C-S-H) through secondary hydration process especially when they are used as partial supplementary cementitious materials (SCM).

1.2 Problem Statements

The waste generated from manufacturing and agro-allied industries has caused a serious threat to the environment. This has necessitated the need to look for a way to convert waste to construction materials. These wastes include palm oil fuel ash (POFA), silica fume, slag, fly-ash, plastic, wood, and papers. The pozzolanic materials (PMs) such as slag, metakaolin, fly-ash (FA), and silica fume (SF) can generate additional calcium-silicate-hydrate (C-S-H) when used as partial supplementary cementitious materials (SCMs). However, in the Kingdom of Saudi Arabia, two of the identified solid wastes are VA and LSP, which are by-products from volcanic eruption and tiles manufacturing industries, respectively. it was reported that about 180,000 km² of VA is available in the western region of Saudi-Arabi (Moufti et al., 2000). Besides, limestone quarrying generates around 20% to 25% LSP (Mageed and AbdelHaffez, 2012). The production of limestone blocks or tiles involves the use of a diamond cutting saw to chip out a large section of the limestone rock into smaller units, these units are further divided into smaller desire pieces as building floor tiles. This cutting process generates powder to the tune of millions of tonnages. These LSP wastes are deposited in landfills or uncontrolled waste fields, the dust particles from the landfills contaminate the air during a strong wind blown, these have resulted in environmental, air, and water pollution leading to severe health hazards such as asthma. Using VA and LSWP as binders in alkaliactivated concrete will lead to a reduction in environmental pollution.

Among the two performance indices for concrete structures are mechanical properties and durability characteristics. VA has been reported to show great resistance to sulfate and acidic attack but it is characterised by long duration setting time. This study looked at the feasibility of synthesizing and also evaluate the durability performance of alkaline activated binder using volcanic ash (VA) and limestone powder waste (LSP). VA and LSP are known to have high silica and CaO contents, respectively. Most of the base materials that have been so far used for alkali-activated binders contain an appreciable amount of Ca, Si, and Al. The skeleton of their binding products includes calcium/sodium-aluminosilicate-hydrate or geopolymer gel (C/N-A-S-H) and calcium-silicate-hydrate (C-S-H) that resembles C-S-H that constitutes the skeleton of the oPC binder. This idea and assumption reinforce the initiation of researching the alkaline activation of the synergy of VA and LSP materials. In which the major factors that affect the mechanical, durability, and microstructural behaviour of the binary blended products were studied in detail. This research has contributed positively to the development of a structural mortar that can be used for repair purposes.

AAM is characterised by very fast early strength development, which has made it suitable for repair work for structures that need to be put in use as in earliest time The determination of mechanical properties for the design of civil engineering structures is of great significance to the construction industry. The compressive strength (CS) of AAM is one of the keys mechanical properties that dictate its suitability for structural purposes. However, the few current existing models for strength prediction only used a few parameters that influenced the compressive strength. This necessitated the modelling of the compressive strength of mortar using machine learning such as support vector regression (SVR) and numerical methods. The choice of SVR for this research is due to the limited data points that characterized the experimental results in determining the properties of AAM.

1.3 Research Questions

The research seeks to address the following questions:

- 1. What is the synergistic effect of VA and LSP on the strength, reaction products, bond characteristics, and microstructure of binary blended alkaliactivated VA/LSP mortar (AAVLM)?
- 2. What is the effect of NaOH concentration, silica modulus, curing temperatures on the strength and microstructural characteristics of AAVLM?
- 3. What is the durability performance of alkali-activated mortar in terms of sulfate resistance and sulphuric acid resistance?
- 4. What estimating models can be used to predict the compressive strength of the development model?.

1.4 Aim and Objective of Research

This research aims to develop alkali-activated binders (AAM) by utilizing the combination of volcanic ash (VA) with limestone powder waste (LSP). The four specific objectives of this research are the following:

- 1. To determine the physical, chemical, and microstructural properties of the based materials volcanic ash (VA) and limestone powder waste (LSP) based materials.
- 2. To determine the effect of alkaline activators parameters and curing temperature on the fresh, mechanical, and microstructural properties of the alkali-activated VA/LSP mortar.
- 3. To examine the durability performance of alkali-activated mortar in terms of sulfate resistance and sulphuric acid resistance.
- 4. To develop a compressive strength prediction algorithm using hybridized genetic algorithms with support vector regression (SVR) method and numerical stepwise regression using key mix parameters as input variables.

1.5 Scope of the Research

The ranges of values used for the optimization of the mix parameters were obtained in the literature.

- Volcanic ash (VA) and limestone powder wastes (LSP) were used as the base materials to develop the alkaline AAM in this study.
- 2. Sodium silicate and sodium hydroxide were used as activators in this study.
- 3. The best heat curing duration was 24 hours.
- 4. The curing of the specimens in this study varied from 25 to 90 °C.
- 5. The optimization of the materials combination was based on;
 - (a) LSP will be added in varying percentages to alkaline activated VA such that the combined ratio.

(b)
$$\left(\frac{LSPW}{LSPW + NP}\right)$$
 varied from (0 to 1) at the interval of 0.2.

- (c) Sodium silicate to sodium hydroxide [NS: NH] was within the range of 0.5 to 1.5 for mortar.
- (d) Alkaline to binder ratio [(NS + NH): BD] was within the range of 0.5 to 0.6.
- (e) Sodium hydroxide molarity [MNH] was varied from 4 to 14M.
- (f) Fine aggregate to binder ratio [FA: BD] was varied from 1.4 to 2.2.
- (g) The optimum free water content (FWC/PMs) ranged from 2 to 10%.
- 6. The durability performance of the selected concrete mixtures was limited to;
 - (a) Sulfate attack (5% Na_2SO_4 , 5% $MgSO_4$ and combination of 2.5% Na_2SO_4 and 2.5% $MgSO_4$).
 - (b) Acid attack (6% H₂SO₄).
- Machine learning (Genetic algorithms and support vector regression (SVR) and empirical modelling were used to model the compressive strengths AAVLM.

1.6 Significance of the Study

This research gave new insightful information about the impact of many factors such as temperature, silica modulus, NaOH concentration on the reaction products, strength development, bond characteristics, and the microstructure of binary blended alkali-activated volcanic ash and limestone powder mortar. The mechanical and durability data from this research will be useful in selecting the appropriate optimum mix which can be used for producing mortar of zero cement and will also contribute to the waste valorization, dump-site land reclamation, low CO₂ footprint, energy consumption reduction, reduction in environmental pollution and addition to more sustainable alternative binders for structural purposes. The estimation model developed will enhance the quick estimation of compressive strength of mortar system to a high degree of precision while it saves valuable time and other material resources.

1.7 Thesis Organization

The thesis is organized into seven different chapters;

Chapter One describes the background, aims and objectives, scope and limitations, and importance and motivation for developing alkali-activated volcanic ash/Limestone powder mortar.

Chapter Two detailed comprehensively reviews the history, development, base materials, and factors affecting the performance of alkali-activated binders. Artificial intelligence modelling and regression.

Chapter Three – In chapter 3 full descriptions of materials and experimental designs were presented.

Chapter Four described the physical and chemical characteristics of VA and LSP. The outcomes on the optimization of key parameters that influenced the strength and microstructures of binary blend alkali-activated mortar.

Chapter five contains the results and discussions on the resistance of the binder to sulfate attack as well as the acidic attack of the synthesized AAVL products.

Chapter six presents the results and discussions with detailed explanations of compressive strength modelling using artificial intelligence and numerical modelling.

Chapter seven summarizes conclusions deduced from this research findings and recommendations for future studies.

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