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CATALYTIC AND PHOTOCATALYTIC performance OF NANOCOBALT FERRITE SYNTHESIZED WITH THE CITRATE PRECURSOR TECHNIQUE

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ABSTRACT:

Supercapacitors and other revolutionary electric energy generation and storage technologies have the potential to significantly improve the environmental situation in densely populated cities by removing the necessity for combustion engines in densely populated areas. Researchers now have a new enlarged platform for finding the correct electrode material capable of providing high power and energy densities thanks to the development of nanomaterials for supercapacitors. The nanostructured electrode material is anticipated to improve the power output and cycle life of the supercapacitor. Many various types of agricultural waste might be seen as an abundant, low-cost carbon source for large-scale manufacturing of graphene-type compounds with added value. We demonstrate how readily available coconut waste (coconut coir and coconut shell) can be effectively converted into reduced graphene oxide by simple catalytic oxidation in a muffle furnace using ferrocene as an efficient and low-cost catalyst. To characterize the structure and morphology of the asprepared materials, XRD, SEM, TEM, and XPS techniques were utilized. The results supported the production of high-quality reduced graphene oxide. The as-prepared graphene oxide electrochemical behavior in 2M KOH aqueous solution was then examined as an electrode material for supercapacitor applications. We observed that the material has strong cycle stability and operates well as an electric double layer capacitor (EDLC). Under visible light irradiation, the rGO from Coconut coir material shown increased photocatalytic activity, destroying around 65% of malachite green dye in 120 minutes. To enhance the electrochemical and photocatalytic capabilities of



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rGO synthesized from coconut coir, we developed novel nickel-based reduced graphene oxide (rGO) nanostructured composites with superior energy storage and photocatalytic properties. rGO nanoflakes were created utilizing a simple hydrothermal technique from negative value coconut coir biowaste and then decorated with functional NiO and NiFe2O4 nanoparticles to create hierarchical functional nanocomposites. Using the synergies formed by using NiFe2O4 nanoflakes and rGO nanoflakes together, the resultant nanocomposites displayed remarkable specific capacitance of 599.9 F/g at current density of 1 Ag-1 and retention rate of 86.5% even after 2000 cycles. Furthermore, the composite displayed photocatalytic degradation efficiency driven by visible light of 96.5%. These findings encourage the multifunctional nanostructured composite's development as a viable material for energy and environmental remediation applications. Recently, there has been a lot of interest in nano/micro-scale Fe-based ferrites with excellent electrochemical capabilities. We developed a simple one-pot hydrothermal technique for producing a variety of cobalt oxides and nanocomposites using biomass waste-derived reduced graphene oxide for alternative spinal metal oxide electrode material as a sustainable energy source. SEM and TEM images confirmed the uniform distribution of metal oxide on the rGO sheets. The functional group and individual components were identified using the FTIR and XPS assays. Composite materials have a higher specific capacitance than pure materials. The rGO spinal metal oxide nanocomposites had the greatest specific capacitance at 1A/g, which was 396 F/g. Furthermore, with a retention of 95.6%, the hybrid supercapacitor has a high cyclic stability of up to 2000 cycles. Malachite green dye was used to evaluate the photocatalytic properties of rGO nanocomposites. Finally, we'd like to emphasize the electrode material's electrochemical and photocatalytic capabilities in connection to the supercapacitor and dye degradation processes. The electrode performance of various metal oxide nanostructures is highly impacted by their microstructure, larger specific surface area, and high electrical conductivity. In 2M KOH electrolyte, CCF (rGO produced from Coconut coir) had the highest specific capacitance of 111.1 F/g while CSF (rGO derived from Coconut shell) had the lowest specific capacitance of 60.2 F/g. They also have 99% stability after 3,000 charge and recharge cycles. Similar studies using



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NiFe2O4 nanoflakes and rGO nanoflakes yielded an excellent specific capacitance of 599.9 F/g at a current density of 1 Ag-1 and a retention rate of 86.5% after 2000 cycles. Furthermore, the composite displayed photocatalytic degradation efficiency driven by visible light of 96.5%. In contrast, rGO spinal metal oxide nanocomposites displayed a maximum specific capacitance of 396 F/g at 1A/g. Furthermore, with a retention of 95.6%, the hybrid supercapacitor has a high cyclic stability of up to 2000 cycles. Using malachite green dye, the photocatalytic results show that rGO/CoFe2O4 exhibited a remarkable efficiency of visible light-driven photocatalytic degradation of 80.8%. At the same time, all of the proposed electrode materials have shown strong electrochemical stability for long-term use.

1 INTRODUCTION

Catalysis is the activity of a catalyst on a process, and a catalyst is a material that raises the pace of the reaction without changing the overall standard Gibbs energy change. Catalysis is not a process that has emerged in recent years. It is a natural process connected with the birth of life. The benefit of a catalytic reaction over other processes is that it occurs at temperatures, provides low highly chosen targets of interest, is less costly, readily regulated, ecologically pure, and so on. Catalysis may be classified as either homogeneous or heterogeneous. The reactant and catalyst are in the same phase in homogeneous catalysis. Acidbase catalysis, enzyme catalysis, and other forms of homogeneous catalysis are examples. The reactant and catalyst

in heterogeneous catalysis are in Metals and separate stages. semiconductors are two examples of catalysis. At the interface between the phases, reactions occur. Catalysts play critical roles in the conversion of waste and raw materials into energy, the reduction of greenhouse emissions, the conversion of monomers into polymers, the creation of material from low-cost sources, and so on. As a result, the chemical manufacturing sector is under immense pressure to find new synthetic processes that are more environmentally benign and more accepted by the catalysis field for the manufacture of commercial goods. Photocatalysis is crucial in this case.



Nano chemistry is a frontier branch of research owing to its many applicability in a variety of sectors. Nanomaterials are used in disciplines such as electronics miniaturization, catalysis, optics, biology, and the energy industry. Furthermore, nano materials produce next-generation computer chips, better insulation materials, tougher and harder Nano materials cutting tools, pollution elimination, high energy density batteries, high-power magnets, highsensitivity sensors, fuel-efficient automobiles. aerospace components with enhanced performance characteristics, longer-lasting satellites, longer-lasting medical implants, ductile, machinable ceramics, large electro chromic display decals, and large electro chromic display decals. Metal nanostructures are used in biological and medicinal applications. As a result of the potential relevance derived from the unique physical features of nano materials, nano research has become the focus of contemporary materials science.

Nano catalysis may aid in the development of catalysts with high activity, selectivity, and stability. These properties are readily obtained by

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adjusting the size, shape, morphology, composition, electrical structure, and thermal and chemical stability of the specific nanomaterial. Although nano catalysts offer various benefits over traditional catalyst systems, isolating and recovering these small nano catalysts from the reaction mixture is difficult. Because of the nano size of the catalyst particles. conventional procedures (such as filtering) are ineffective. This constraint jeopardizes the economics and long-term viability of these nano catalytic techniques. To address this problem, magnetic nano particles have emerged as a feasible alternative; their insoluble and paramagnetic character allows for simple and fast separation of the catalysts from the reaction mixture using an external magnet.

Nano particles are often characterized as materials with one or more dimensions smaller than 100 nm and, as a consequence, exhibit unique characteristics that vary from their bulk equivalents or atomic / molecular components [1]. Nano particles have a long history, dating back to ancient Egypt, Iran, and Rome, when colloidal



gold was utilized for ornamental decoration [2]. The most renowned specimen of this kind is the 4th century Lycurgus Cup, which is now housed at the British Museum. However, it was not until 1857 that Michael Faraday made red gold sols by reducing gold in solution with white phosphorus [3]. He referred to them as split metals and demonstrated that these metal sols were thermodynamically unstable and hence needed to be stabilized against aggregation. The twentieth century saw several improvements in colloidal gold production technologies [4]. Bredig employed an electrochemical approach [5], whereas Zsigmondy invented the seeding method [6], for which he was awarded the Nobel Prize in 1925. This was followed by Turkevich's seminal work with citrate-stabilized sols [7] and Schiffrin's with Brust and work monolayer-protected clusters [8]. Because of their tiny size and thus high surface to volume ratio, nano particle materials have recently been the subject of active study. As a result, unique features such as superparamagnetism, morphological tuneable optical properties, and stable photo luminescence have emerged [9]. The

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subject of nano science has grown at an exponential rate year after year owing to novel synthetic methods and the availability of new characterization techniques [10].

2 LITREATURE SURVEY

researchers been Some have compensated to investigate the impact of environment on the the arsenic adsorption capacity of magnetite Fe3O4 micro particles [81, 84]. Among iron oxides, magnetite nano particles have the greatest saturation magnetization of 90 emu g-1. Magnetite nanoparticles may therefore be utilized to absorb arsenic ions before magnetic decantation. Arsenic activity has been found in several iron oxides and hydroxides. These compounds, however, have substantially lower magnetic characteristics than magnetite. Magnetite oxidation is discovered, which results in a decrease in saturation magnetization. The group discovered that replacing Fe2 + in Fe3O4 with a little quantity of Co2+ or Ni2+ may increase the compound's oxidation resistance [85]. Under atmospheric circumstances, oxidation resistance is a crucial element in arsenic removal. The



arsenic adsorption capacity of Fe1xCoxFe2O4 (Co-ferrites) and Fe1yNiyFe2O4 (Ni-ferrites) micro particles (x, y = 0, 0.05, 0.1, 0.2, 0.5) is investigated in this study.

As a durable, easily accessible, high heterogeneous surface-area catalytic support, functionalized magnetic nano particles have emerged as feasible replacements to traditional materials. [86] They have the extra benefit of being magnetically separable, which eliminates the need for catalyst filtering once the reaction is complete. Furthermore, there is an urgent need to find non-precious metal catalysts for the hydrogenation of alkynes that are less costly and readily accessible. [87] Developing greener and more sustainable methods for organic transformations [88] and nanomaterials. [89]

ferrite (CuFe2O4)is Copper an spinel ferrite MFe2O4 important because it undergoes phase transitions, changes semiconducting properties, exhibits electrical switching, and tetragonality variation when subjected to different conditions, in addition to

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interesting magnetic and electrical properties with chemical and thermal stabilities [112]. It has several uses, including gas sensing [113], catalytic applications [114-116], Li ion batteries density magneto-optic [117], high recording devices. color imaging, bioprocessing, magnetic refrigeration, and ferrofluids [112,118]. Furthermore, CuFe2O4 is significant due to its high electric conductivity, thermal stability, and catalytic activity for O2 evolution from the alumina-cryolite system employed in aluminum synthesis [119]. CuFe2O4 has been found in tetragonal and cubic forms. Cuferrite crystallizes in a tetragonal form during gradual cooling, with a lattice parameter ratio c/a of roughly 1.06. Cu-ferrite's tetragonal phase features an inverse spinel structure, with practically all Cu2+ ions occupying the octahedral sub lattice, whilst Fe3+ ions split evenly between the tetrahedral and octahedral sub lattices [105]. Due to John-Teller distortion, the tetragonal structure is stable ambient at temperature and transitions to cubic phase only at temperatures of 360 °C and higher. The magnetic characteristics are closely connected to the distortion. The cubic structure has a greater



magnetic moment than the tetragonal structure because there are more cupric ions (Cu2+) occupying tetrahedral sites in the cubic structure than in the tetragonal structure [106]. Nano-sized copper spinel ferrites exhibit remarkable features when compared to their bulk analogs and have received a lot of research over the past decade due to their prospective uses. They may be produced using a number of processes, including solid-state reaction [107], mechanochemical [108], sol-gel [109], co-precipitation [110], combustion synthesis [111], polyol approach [112], and micro emulsion hydrothermal route [113]. The majority of the preceding path resulted in the creation of tetragonal copper ferrite. To the best of my knowledge, the synthesis of cubic ferrite powders by the copper hydrothermal approach is a seldom described topic. The association between the magnetic and catalytic characteristics. well as as the microstructure of the cubic copper ferrite generated by the hydrothermal process, requires more research. Because of the potential uses of ferrites, significant effort has been focused on the synthesis of ferrites from industrial

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istes. Copper ferrite CuFe2O4 is made by ball milling copper sludge collected from a filter press of an iste-water treatment process in a surface finishing factory with analytical grade iron oxide powder [114]. Ultrasonic-assisted chemical co-precipitation is used to create nano-sized magnetic particles from wasted pickling liquors [115].

3 METHODOLOGY

This chapter examines the various methods of synthesis of bulk materials and the many experimental techniques used for characterization. Ceramic, solgel, citrate precursor, combustion, coprecipitation, and other processes are available for the manufacture of oxide materials [1, 2]. The co-precipitation approach is the most often utilized method. The coprecipitation approach was used to manufacture several Nitrate compounds in the current investigation. In addition to this approach, ceramic, sol-gel, citrate precursor, and combustion were utilized in the production of nano crystalline cobalt ferrite. Metal oxides that decompose into reactive products are employed as precursors in the co-precipitation



process. Precursors must be very pure and in fine powder form, with sufficient reactivity [3]. The precursors are precisely weighted in the requisite molar proportions.

To achieve a complete reaction in the shortest time and at the lowest possible temperatures, mixing of the component cations on an atomic scale is required. Compound precursors achieve this goal, but the stoichiometry of the precursors does not always strictly coincide with the stoichiometry of the desired product

The goal of this research is to employ a unique and cost-effective technique of preparing Cobalt ferrite using the citrate precursor approach in order to at accomplish sintering lower temperatures. The citrate technique is a kind of sol-gel method for metal oxide production that use citric acid as a complexing agent [10]. It is a faster process than the sol-gel method that uses metal alkoxides. In this procedure, the relevant metal salts and citric acid are dissolved in a suitable solvent (water), and the resultant solution is heated to allow the solvent to evaporate. Because of the constant elimination of the solvent during evaporation, the solution gets

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more viscous. Finally, a gel is formed, and calcination at an appropriate temperature produces the matching metal oxides. Because of the molecular level mixing of the component metal ions, the citrate gel technique is suited for the production of mixed metal oxides. Furthermore, the process is well suited for producing nano crystalline materials of various sizes and shapes by varying the quantity of metal ions, metal ion citric acid. calcination ratio to temperature, and so on. The citrate gel approach has been extensively employed for the production of nanoparticles of various mixed metal oxides including more than one metal, such as ferrites, Perovskites, and cobalt ferrite [11-14].

For the investigation, Cobalt Ferrite with the general formula CoFe2O4 was used. In a 1:1 molar ratio, aqueous solutions with stoichiometric quantities of cobalt nitrate and ferric citrate were treated with citric acid. To complete the process, the pH of the solution was raised to 7 by adding ammonium hydroxide. The solution was gently evaporated to dryness over a ten to twelve hour period. The solution's viscosity and color varied as it transformed into a puffy, porous



dry gel. After the solvent is removed, the dried precursor undergoes a self-ignition process, yielding a very fine powder known as as-synthesized powder.

4 EXPERIMENTS & RESULTS

The use of eco-friendly ferrites [1-4] for effective solar energy use is becoming more common in visible light photo catalysis research. Ferrites are well recognized for their many uses in catalytic, magnetic, and electronic materials [5, 6], however there have publications been few their on photocatalytic applications [7, 8]. Unlike TiO2, ferrites have the benefit of having desired optical absorbance for low energy photons (hv 2 eV) as well as a well-suited electrical for structure photocatalytic applications. Many efforts have been made to create the narrow band gap ferrite semi conducting material that exploits the considerably broader visible area in order to fully utilize solar energy. Some recent findings may be crucial indications of the potential of spinel ferrites for visible light photocatalytic applications. For example, Jang et al. [9] and Jung et al. [10] proved that ZnFe2O4 and CaFe2O4 systems, respectively, are beneficial for

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solar photocatalytic degradation of contaminants. Similarly, CaFe2O4:MgFe2O4 and ZnFe2O4:SrTiO3 have been found to be efficient beneficial for and photocatalytic water splitting in homo [11] or hetero [12] composite ferrite systems. Because of their ecologically acceptable features, availability, cheap cost, and benign nature, iron oxide semiconductors and other iron-based catalytic and photocatalytic materials are particularly appealing. As a result, irondouble based perovskites and perovskites are being explored for prospective improvements in iron's catalytic characteristics. However, there are limited publications on the photocatalytic activity of ferrite type compounds such as MFe2O4 [13], LaFeO3 [14], ZnFe2O4 [15], and NiFe2O4 [16], including hydrogen production through water splitting. Furthermore, research into iron-based rare earth perovskites and the effect of certain rare earth metals on the electrical and photocatalytic characteristics of ferrites is of interest. Although Oudenhoven et al. have beautifully explored the influence of rare earth elements via spinal systems, there is



some doubt concerning the behavior of such materials, which necessitates more research. With this as a backdrop, ironbased PrFeO3 perovskite was synthesized utilizing various ways, and capabilities its photocatalytic for hydrogen production through alcoholassisted water-splitting process were investigated. Our lab has previously reported a LaFeO3 type perovskite with relatively high photocatalytic hydrogen production [17], and we are now expanding this research to praseodymium ferrite, which has been intensively explored for its ferroelectric and ferromagnetic characteristics [18,19]. To the best of our knowledge, PrFeO3 perovskite has not been investigated for thoroughly photocatalytic hydrogen production through water splitting.

Typically, photodegradation processes in heterogeneous occur systems. Recovery and re-use of photocatalysts after deterioration are critical for longprocess management. The term incorporation of magnetic nano particles into a range of solid matrices enables the combining of well-known approaches for catalyst heterogenization with

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magnetic separation techniques. Cobalt ferrite (CoFe2O4) with an inverse spinel structure exhibits ferrimagnetism caused by an antiparallel magnetic moment between Fe3+ ions at tetrahedral sites and Co2+ ions at octahedral sites. Titania-coated cobalt ferrite. a magnetically separable photocatalyst, was created using magnetic nano particles of cobalt ferrite [21]. Despite the fact that CoFe2O4 has a band gap of 1.68 eV, [22] to the best of our knowledge, little study has been undertaken on its photocatalytic activity when exposed to visible light. Pure cobalt ferrite is an inert photocatalytic substance. Despite this, it is feasible to increase the effectiveness of photoinduced charge separation by coupling CoFe2O4 nano particles with another semiconductor (e.g., graphene sheets), resulting in excellent photocatalytic performance. The construction of a NiFe2O4-graphene photocatalyst and its remarkable performance in the photocatalytic degradation of MB in the presence of hydrogen peroxide under visible light irradiation was recently reported. [23] It is commonly understood that hydrogen peroxide is produced by a process that uses energy



and/or other chemical resources. As a result, designing magnetically separable graphene-based photocatalysts with high catalytic activity in the absence of hydrogen peroxide is of interest.

A 100 ml of 10-4M aqueous solution of sample was obtained in a beaker placed under the light source at room temperature. Before starting the photocatalytic process, the liquid was agitated for 30 minutes with a cobalt ferrite - graphene (10%) composite catalyst to reach adsorption equilibrium. Following the irradiation, the sample solution was extracted, centrifuged to remove the catalyst particles, and suitably diluted in order to assess its absorbance/concentrations at the characteristic wavelength. Its deterioration percentage is computed based on this. The absorb ance/ concentrations of dyes were determined using a UV-Visible Spectrophotometer. Percent of Degradation = $Co - C \times 100 /$

Since the past three decades, researchers all over the world have been progressively developing ultrasound aided organic synthesis for the synthesis of organic compounds. Ultrasound

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irradiation provides an alternate energy source for organic processes that are typically carried out by heating. The main advantages of ultrasonic irradiation include faster reaction rates, purer products, simpler manipulation, energy conservation, waste reduction, and this is more suitable for approach incorporating green chemistry ideas [1-5]. The development, expansion, and collapse of acoustic bubbles in the reaction media is how ultrasoundassisted reactions work. These immediately contribute to reducing response times and enhancing yields [6]. Furthermore, a large number of reactions may be carried out in less time, with a greater yield due to gentler reaction conditions under ultrasonic irradiation [7-9]. Many homogeneous and heterogeneous reactions may be carried out easily by sonication, resulting in higher yields and selectivity Ultrasound [10-12]. accelerates chemical processes in a solution by generating cavitation in the form of tiny bubbles. The expansion and contraction of these micro bubbles cause energy to be transferred and focused from the macro scale motion of the acoustic transducer to the micro size vapors



within the bubbles. During collapse, the within bubble vapour phase the generates very high pressures on the scale of hundreds of atmospheres and extremely high temperatures on the order of thousands of degrees Kelvin. As a result, extremely reactive free radicals are formed. This one-of-a-kind energy concentrating technique allows chemicals to react in an aqueous solution. This technology's potential uses span from the degradation of environmental contaminants to medication production for medicinal therapy. The amplitude and frequency of an applied sound field, temperature, surface tension, vapour pressure, gas concentration and nuclei density of the solution, as well as vessel and probe design, control sonochemistry [13]. The of intimate connection these characteristics, as well as the micro bubbles' tiny size and rapid oscillation frequency, and the low concentration of species inside each one bubble, make experimental theoretical and examinations of sonochemistry exceedingly challenging. Nonetheless, а variety of examinations from diverse viewpoints have been conducted, each of which has provided significant insight

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into this interesting occurrence. It is shown that nano nickel ferrite materials have been employed as magnetically separable and reusable heterogeneous catalysts for the production of, unsaturated ketones through the allylation of acid chlorides with allyl bromides. The reaction of allyl halides with different acid chlorides is carried out at room temperature in tetra hydrofuran (THF) reflux under conditions in the presence of nano ferrite particles. The current technology is ecologically friendly and produces excellent results. Using a powerful neodymium magnet, the catalyst was removed from the reaction media and reused numerous times without losing much catalytic activity. The reaction mechanism and product characterisation are provided.

5 CONCLUSION

A composite material is made up of two or more constituent materials that do not include any metals and have entirely separate chemical and physical characteristics. Actually, it is a combination in which the constituent components are distinct and independent from one another at the macroscopic



level. When at least one of the constituent materials has a physical dimension in the nanoscale scale domain, a composite material is said to be nanocomposite. In practice, hybrid nanocomposites are classified into two types: organic and inorganic. One kind is inorganic-organic nanocomposite, in which the organic component (e.g. conjugated polymer) is deposited in an inorganic template or substrate (e.g. silica, alumina, etc.). Organicinorganic nanocomposite is another form in which nanoparticles of oxide inorganic materials are placed in a suitable polymer matrix. As a result, there are continuous two phases: and discontinuous. The continuous phase of organic-inorganic nanocomposites is formed of organic components, while the discontinuous phase is comprised of nanoparticles.

Furthermore, it requires a lengthy processing time. As a result, fabricating a device using bulk single crystal is prohibitively costly. So, from a business standpoint, there is always the curiosity of having a different approach. In compared to bulk single crystal inorganic oxide materials, device

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components made of organic-inorganic nanocomposite materials offer all of the benefits of organic materials, such as great mechanical flexibility, facile production, and hence cheap cost. Furthermore, such nanocomposites may be employed in a variety of applications where the bulk inorganic equivalent has failed. These hybrid materials have the potential to be useful in small integrated Organic-inorganic photonic devices. nanocomposites have been created in this chapter by distributing the generated LN nanoparticles into a polymer mtrix, poly(methyl methacrylate) (PMMA), and the entire composite is referred to as LN/PMMA nanocomposite. Various LN/PMMA nanocomposites have been prepared to investigate their linear and nonlinear optical properties in order to comprehend the collective behavior of LN the nanoparticles and their functionalities in relation the to nanocomposite thickness, nanoparticle concentration, and orientations in the presence of an externally applied electric field.

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