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Article

# A Review of Analytical Approaches for Detecting Microplastics and Their Degradation Products in Biological and Environmental Samples

**Arshed AbdAli Shihad[1], Fadhel Ibrahem Aljabari[2]**

1. Ministry of Education, Al-Qadisiyah Education, Dewaniyah, Iraq
2. Department of chemistry, college of sciences for women, Baghdad University
Correspondence: [1]arshedalmansori@ec.edu.iq, [2]Fadhil.i@csw.uobaghdad.edu.iq

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**Abstract:** This study investigated the key environmental, socio-economic, and infrastructural factors influencing the current ecological challenges in the target region. Field observations, supported by quantitative and qualitative assessments, revealed significant pressures on natural resources caused by population expansion, weak waste-management systems, and limited regulatory oversight. Soil, water, and air quality indicators demonstrated varying degrees of deterioration, reflecting the cumulative impact of industrial activities, agricultural misuse, and insufficient environmental planning. The analysis further highlighted the absence of long-term sustainability strategies, fragmented institutional coordination, and low community awareness as major obstacles to environmental protection efforts. Statistical interpretation of collected data confirmed strong correlations between anthropogenic activities and the recorded decline in ecosystem performance. A number of targeted interventions were discussed, emphasizing the need to strengthen environmental governance, adopt integrated resource-management policies, and enhance scientific monitoring programs. Overall, the study provides an evidence-based evaluation of current environmental conditions and identifies a set of practical recommendations that can support decision-makers in developing more effective mitigation and adaptation frameworks. These findings contribute to a clearer understanding of the environmental transformations occurring in the region and underscore the urgency of implementing sustainable solutions

**Keywords:** Environmental Degradation, Natural Resource Management, Anthropogenic Activities, Environmental Governance, Sustainable Development

## Introduction

Microplastics have become a ubiquitous and persistent group of environmental pollutants that is of international concern because of their omnipresence, persistence, and possible ecological and human concerns [1]. Microplastics can be defined as plastic particles that are less than 5 mm in size; they can be primary particles that are purposefully created to be used in the industrial or cosmetic sector, or secondary particles that are broken off. With the ever-increasing plastic use on the planet, microplastics have been detected globally, and in increasing numbers, in different environmental compartments like in oceans, rivers, soils and sediments, and aerosols in the atmosphere, and in various biological matrices such as seafood, mammalian tissues, and even in the blood and placenta of humans [2]. This large-scale dispersion highlights the seriousness of the situation, whereby there is a need to come up with advanced analytical instruments that can effectively identify, describe, and measure these pollutants [3].

The increasing alarm is not based on the microplastic particles alone, but on them and their degradation products, as well as their related chemical additives. Plastic polymers begin to degrade progressively due to exposure to sunlight, mechanical stress, oxidative conditions, and microbial activity to produce nanoplastics, as well as a broad range of chemical byproducts, including monomers, oligomers, bisphenols, phthalates, and volatile organic compounds. These small and chemically reacting substances can have a better bioavailability and toxicity than larger microplastic particles. Toxicological, ecotoxicological, and biotoxicological analyses of the environmental fate, transport, and biological interactions of microplastics and their degradation products demand both strong and sensitive methods of analysis that are able to deal with the complex environmental and biological sample matrices [4], [5].

Irrespective of the remarkable progress in the research on microplastics that has been achieved in the last 10 years, there are still analytical difficulties. Microplastics are diverse in size, density, shape, polymer type, and surface chemistry, making extraction, identification, and quantification tasks difficult. Proteins, lipids, and cell debris commonly occur in biological samples and can become a nuisance to detection methods, whereas environmental samples can be highly variable in terms of their inorganic and organic particulates, which must be removed effectively. There are still no standardized protocols regarding sample preparation and analysis, and thus inconsistencies between the laboratories, which prevent reliable cross-study comparisons [6], [7].

The methods used to detect microplastics include a wide range of methods, including spectroscopic, microscopic, chromatographic, and thermal. New technologies like asymmetrical flow field-flow fractionation, nano-FTIR, single-particle ICP-MS, and AI-controlled imaging have broadened the set of detectable particles and more complex degradation products, since they allow characterization of smaller particles and more complex degradation products. Nevertheless, all of the techniques have their peculiar advantages and disadvantages in terms of sensitivity, resolution, throughput, and cost [8], [9].

This review will offer a thorough and more recent assessment of the analytical procedures that are used nowadays in order to detect microplastics and their degradation products in environmental and biological samples. Through the comparative analysis of conventional methods and emerging technology, the review presents recent innovations, key issues, and future insights that should be undertaken to improve the accuracy, standardization, and reproducibility of microplastic research [10], [11].

## Microplastics Characterization: Physical and Chemical Properties

In the characterisation of microplastics, it is important to have good knowledge of their physical and chemical properties because these attributes indicate how they behave under environmental conditions, how they interact with biological systems, and how they react to the methods used in the analytical process. Microplastics are highly heterogeneous regarding size, morphology, density, polymer type, and surface chemistry. They usually measure a few nanometers to a few millimeters in size; smaller particle fractions like nanoplastics are more difficult to analyse because of detection resolution restrictions and have the tendency to bioaccumulate more. Shapes can be as small as fragments, fibers, pellets, granules, and films, each type having an impact on mobility, degradation mechanism, and sorption propensity of contaminants [12].

Polymer composition is an important feature, and popular polymers are polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and polyethylene terephthalate (PET). The degradation rates, spectral signatures, and detectability of these polymers under spectroscopic techniques are determined by the chemical structure and crystallinity of the polymer. Moreover, density is also a decisive factor that influences the distribution of microplastics in aquatic environments. Dense polymers are more likely to float, whereas more massive ones can be found in sediments [13].

Other properties of surfaces, such as roughness, oxidation stage, and biofilm formation, are also significant. Processes of weathering bring in cracks, pores, and functional groups, including carbonyl and hydroxyl groups, that change the optical and chemical features that are measured by instruments like FTIR or Raman spectroscopy. All these parameters affect the selection of the analysis method and determine the intricacy of the process of sample preparation [14].

**Table 1.** Common Physical and Chemical Characteristics of Microplastics Relevant to Analytical Detection.

| Property | Description | Analytical Implications |
|---|---|---|
| Size | Nano- to millimeter scale | Smaller sizes require advanced imaging and high-resolution tools |
| Shape | Fibers, fragments, pellets, films | Shape influences sedimentation and detection efficiency |
| Polymer Type | PE, PP, PS, PVC, PET | Determines spectral signatures in FTIR/Raman |
| Density | 0.9–1.4 g/cm$^3$ depending on polymer | Affects separation and flotation methods |
| Surface Chemistry | Weathered surfaces, oxidation groups | Influences adsorption, identification, and degradation behavior |

## Sample Collection and Pre-treatment Techniques

The proper sampling and pre-treatment methods are vital in the accurate detection of microplastics in both the environmental and the biological matrices. The integrity and representativeness of the results are directly affected by each step of the sample collection process, i.e., the sampling sites selection to prevent contamination. Environmental samples such as water, sediments, soils, and atmospheric particulates must be collected using standardized procedures to reduce the external contamination of plastics. In the case of water samples, stainless steel or glass sampling equipment is desirable to eliminate any interference with plastic, and coring or grab methods are often necessary to maintain vertical profiles and distribution of particles in soil and sediment samples [15].

Pre-treatment is used to separate microplastics in complex sample matrices and to remove other non-plastic organic or inorganic matter, which could be a source of interference in subsequent analysis. One of the most widely utilized methods of aquatic samples includes filtration, whereby a mesh is used with a corresponding size that matches the range of particles of interest. High-density salt solutions to be used include zinc chloride or sodium iodide to provide a density difference between the floating and sinking plastic particles. This technique is especially effective in sediments, whereby mineral particles can conceal the existence of microplastics [16].

There are other challenges in biological samples because they are highly organic. A softer substitute to chemical treatments is enzymatic digestion with proteinase K, lipase, cellulase, or chitinase to preserve the polymer integrity. Nevertheless, the use of hydrogen peroxide, potassium hydroxide, or nitric acid as a reagent to carry out chemical digestion is still popular because it is a highly efficient way to digest polymers, even though it may cause some alterations in sensitive polymers. To achieve particle purity before analysis, the rinsing, drying, and repeated filtration steps are frequently necessary [17].

Strict control of contamination must be performed during all pretreatment procedures. Airborne contamination of microfibers is reduced by the use of non-plastic equipment, a clean laboratory, procedural blanks, and handling procedures. Finally, the quality of the sample collection and pretreatment has a direct influence upon the sensitivity and accuracy of the analysis, which is why the standardisation and validation of the procedures should be developed and implemented in laboratories [18].

**Spectroscopic Analytical Techniques**

The foundational method of microplastic detection and identification is spectroscopic methods as a way of producing polymer chemical fingerprints. Among them, Fourier Transform Infrared Spectroscopy (FTIR) and Raman spectroscopy are the most commonly used ones. FTIR identifies the vibrations of molecular vibrations present in a sample and results in recognizable absorption spectra that enable the accurate identification of a polymer. Micro-FTIR, which has imaging capabilities, allows particle analysis to 1020 µm and is most effective in the analysis of environmental samples with heterogeneous plastic fragments. But FTIR has drawbacks of resolution to the size of particles and can be affected by oxidation caused by weathering and changes in spectral signature [19].

Raman spectroscopy has complementary functions, with the potential to map chemical distribution with high resolution, as well as the possibility to identify smaller particles, down to submicron sizes. The latter is especially beneficial due to the fact that Raman requires little sample preparation and can even penetrate colored or opaque particles, which can be rather problematic with FTIR. However, a significant limitation is still present in fluorescence interference, particularly with biological samples that contain high pigments or organic residue.

The near-infrared (NIR) spectroscopy is a relatively new tool that is considered in the role of rapid screening. Even though this is not as sensitive as FTIR or Raman, NIR is appropriate in high-throughput and real-time analysis as well as industrial use. Bulk characterization of plastic waste streams is possible given their capability to infiltrate deeper into materials [20].

Microscopy and automated image processing tools are also being integrated with spectroscopic methods, which enable researchers to obtain more precise counts of the particles, shape identifications, and polymer classifications. These hybrid models are much more effective in detection capacity and minimize the human error factor, but demand advanced instrumentation and high-level computing resources [21].

**Table 2.** Comparison of Major Spectroscopic Techniques Used for Microplastic Analysis.

| Technique | Detection Size Range | Advantages | Limitations |
|---|---|---|---|
| FTIR/micro-FTIR | ~10–20 µm | Accurate polymer ID; imaging capability | Limited resolution; affected by oxidation |
| Raman | Sub-micron to >10 µm | High resolution; minimal sample prep | Fluorescence interference; costly equipment |
| NIR | >100 µm (typically) | Rapid screening; suitable for bulk analysis | Lower sensitivity; limited polymer discrimination |

**Microscopy-Based Techniques**

Microscopy-based methods are deeply involved in the physical characterization of microplastics, especially in terms of morphology, size distribution, and surface topography of particles. Although microscopy is not a tool that can be used to identify a chemical per se, it cannot be replaced as a complementary method to spectroscopy. The simplest method is optical microscopy, which is used to observe particles with dimensions greater than about 50, 100, 100. It is most commonly employed as a primary screening method because it is cheap and a simple procedure. Nonetheless, it does not have the resolution or precision needed to identify particles that are in the nanoscale, nor does it have a limit due to the subjectivity of operators [22].

With a much higher level of resolution, Scanning Electron Microscopy (SEM) would allow one to see the surface of microplastics in greater detail, including cracks, wear, and biofilm structures. SEM imaging has been useful in determining the process and mechanisms of degradation of microplastics as well as their interactions with the environment. SEM can be used in conjunction with Energy

Dispersive X-ray Spectroscopy (EDS) to provide information on elemental composition, and it can be used to determine the presence of contaminants and additions on the surface, but not the type of polymer [23].

The Transmission Electron Microscopy (TEM) has a resolution of at least the nanometer scale, which is why it can detect nanoplastics and analyse their interior structure. TEM has also found wider application in investigations of higher degradation states, whereby polymer fragments are in a nanoscale size. Nonetheless, TEM is characterized by a long preparation of samples and is quite expensive, and its usage is restricted to regular monitoring.

Another instrument that comes in handy is fluorescence microscopy, especially when the microplastics are stained using such stains as Nile Red, which selectively attaches to the surface of hydrophobic polymers. The method allows for quick identification of microplastics in complicated samples, including biological tissues or wastewater. Recent developments in machine learning algorithms and automated image processing methods have greatly enhanced the process of particle recognition, classification, and quantification, and minimized human bias and augmented throughput [24].

As such, microscopy continues to be a crucial cornerstone to research on microplastics, as it gives an important visual base that supplements chemical analysis and the overall effectiveness of detection methods [25].

## Chromatographic Techniques for Detecting Degradation Products

Chromatography is also necessary to study chemical additives, monomers, or degradation products emitted by microplastics in the environment during weathering or biological engagement. In contrast to spectroscopic and microscopy-based techniques, which are mainly concerned with physical and structural identification of the particles, chromatography is highly sensitive and selective to small organic molecules that leak out of plastics. These byproducts of degradation, which include bisphenol A (BPA), phthalates, and styrene compounds, have high toxicological risks, which underscores the need to achieve high-quality detection [26].

Mass spectrometry (GC-MS) combined with gas chromatography has remained one of the most potent techniques for identifying volatile and semi-volatile compounds produced as a consequence of thermal or photochemical degradation. GC-MS is especially useful in the detection of styrene monomers, aromatic hydrocarbons, and additives of polystyrene, polyvinyl chloride, and polyethylene. The Pyrolysis- GC-MS (Py-GC-MS) also has an advantage in that it can directly decompose the chains of polymer under high temperatures and therefore identify the type of polymer used without any prior extraction. Nonetheless, it is sometimes hard to interpret, and it also needs specialized equipment [27].

Liquid chromatography in tandem mass spectrometry LC-MS/MS has been extensively used in the quantification of non-volatile degradation products and additives, including BPA, phthalates, and plasticizers. It is sensitive and can be used in aqueous matrices, which is why it is an ideal tool with biological samples since the concentrations of the compounds are usually very low. Fluorescence or UV screening Polymer additives in water and sediments often are screened by using high-performance liquid chromatography (HPLC) in conjunction with fluorescence or UV detection [28].

Although effective, chromatographic methods have some challenges, such as requiring a long sample purification process, interference of the matrix, and the unavailability of reference standards of newly forming degradation products. The development of methods, calibration, and validation is still central to making quantification and comparability of studies accurate [29].

**Table 3.** Major Chromatographic Techniques Used for Analyzing Microplastic Degradation Products.

| Technique | Target Compounds | Advantages | Limitations |
|---|---|---|---|
| GC-MS | Volatile and semi-volatile compounds | High sensitivity; detailed structural analysis | Requires derivatization; complex spectra |

| | | | |
|---|---|---|---|
| Py-GC-MS | Polymer fragments, monomers | Direct analysis of polymers | Costly; thermal degradation may alter products |
| LC-MS/MS | BPA, phthalates, oligomers | Excellent for biological samples; high accuracy | Requires sophisticated sample preparation |
| HPLC-UV/FLD | Additives, dyes, plasticizers | Good for screening and routine analysis | Lower sensitivity compared to MS |

### Thermal Analysis Techniques

Thermal analysis methods offer a useful understanding of the behavior of microplastics and their degradation mechanisms owing to the evaluation of the variations in their physical and chemical properties under carefully regulated heating environments. The techniques are specifically helpful when it is impossible to identify or determine the amount of polymer by spectroscopic or microscopic methods only. Thermal methods help researchers to identify typical thermal changes, decomposition temperatures, and mass loss studies that are suggestive of certain types of polymers [30].

One of the most commonly used thermal techniques is the Thermogravimetric Analysis (TGA). It is used to determine weight loss in relation to temperature, providing data on thermal stability and degradation of polymers. TGA is able to distinguish between polymer types by their decomposition temperatures and can also give semi-quantitative results of microplastic levels in complex matrices, i.e., sediments or sludge. Combined with mass spectrometry (TGAMS) or Fourier Transform Infrared Spectroscopy (TGA-FTIR), evolved gases produced during polymer degradation may also be further analyzed to determine individual decomposition products to improve the analytical resolution [31].

Another significant method of determining transitions is Differential Scanning Calorimetry (DSC), which is used to measure temperature changes in melting, crystallization, and glass transition. Each type of polymer has its own thermal fingerprint, and this helps differentiate between polyethylene, polypropylene, and polystyrene among other polymers. DSC can also serve to determine the degree of weathering since degradation of the environment can tend to change polymer crystallinity and thermal characteristics [32].

Methods based on pyrolysis supplement TGA and DSC as they allow direct thermal degradation of polymers to smaller scales that can be analyzed by chromatographic or mass spectrometric methods. The method is especially robust in the case of mixed polymer samples or nanoplastics, which can be challenging to isolate by physical methods.

A combination of thermal analysis techniques is helpful in obtaining solid evidence on the identity of polymers, the process of their degradation, and thermal stability. Their combination with spectroscopic or chromatographic systems makes them much more detectable and benefit a more extensive analysis of microplastic pollution in the environment and biological samples [33].

### Emerging and Advanced Analytical Approaches

The rising sophistication of microplastic pollution and the awareness of nanoplastics have led to the emergence of sophisticated analytical methods out of the shortcomings of traditional spectroscopic, microscopic, and chromatographic methods. Of these, Asymmetrical Flow Field-Flow fractionation AF4 has become an effective instrument for segregating micro- to nanoscale plastic particles in hydrodynamic size. Since there are no stationary phases required to carry out high-resolution fractionation, AF4 eliminates the loss and risks of contamination of samples. In combination with sensors like multi-angle light scattering (MALS) or ICP-MS, AF4 can also give information on particle size distribution and elemental composition in detail, and thus is especially useful in characterizing nanoplastics that are otherwise inconvenient to characterize using conventional microscopy and spectroscopy [34].

Nano-FTIR spectroscopy is another breakthrough technology that is a combination of nanoscale imaging with vibrational spectroscopy that can achieve chemical identification at resolutions of 20 nm. This is an intermediate between Raman and electron microscopy, allowing high-resolution mapping of surface chemistry and giving information on degradation mechanisms, biofilm formation, and interaction between particles and cells. Nano-FTIR is also restricted by its cost and availability, making it only accessible to use in specialized research labs despite its capabilities [35].

Single-particle ICP-MS has also become popular in the process of micro- and nanoplastics detection using elemental analysis. Although plastic polymers do not have any metal signatures, additives, pigments, and surface contaminants can be detected with the help of this technique. It is especially helpful in the observation of the environmental transport processes and determination of the interaction of microplastics and heavy metals [36].

Microplastic detection is being revolutionized using AI and methods of machine learning to provide automated recognition of particles, classification, and quantification. State-of-the-art image-processing algorithms can reduce operator bias by a large factor and can improve throughput, whereas deep-learning models can distinguish between polymer types, be it by spectral pattern or morphology. All of these developing methods are a step in the right direction of combining high-resolution analytical methods with computational intelligence, providing new opportunities to the precise, fast, and scalable detection of microplastics and their degradation products [37].

## Environmental and Biological Implications of Microplastics and Their Degradation Products

The presence of microplastics and degradation products is a complex threat to both environmental and biological systems because of their persistence, chemical reactivity, and the capacity to accumulate in food webs. Microplastics in aquatic ecosystems may be consumed by a broad set of organisms, such as zooplankton, fish, mollusks, and marine mammal organisms, and cause the physical blockage, altered feeding behaviors, and increased energy expenditure. Being tiny in size, they can be easily transferred across biological barriers, which allows particles to get into digestive tissues and circulate within the systems of the body. Nanoplastics have an even greater capacity to translocate, are able to pass through cell membranes, and may accumulate in organs, as well as disrupt cellular functions like oxidative balance and gene expression [38].

Chemical degradation products also add to toxicity, in addition to the physical presence of polymer particles. The bisphenols, phthalates, flame retardants, and other additives leak out of the microplastics during environmental or physiological environments and have been implicated in endocrine and reproductive dysfunction and immunotoxicity in all sorts of species. These substances can be trophically magnified, exposing top-level organisms to a greater risk and possibly making it to human beings by eating seafood. Also, environmental pollutants, including heavy metals, polycyclic aromatic hydrocarbons, and pesticides, can be adsorbed on weathered microplastics, which are vectors for carrying toxic compounds through the ecosystems [39].

On land, microplastics may change the soil structure, decrease the ability to retain water, and have an impact on the composition of microbial communities. They can affect the growth and reproduction of soil organisms such as earthworms and insects, causing more ecological imbalances. Another topic under development is atmospheric microplastics because they may be inhaled, deposited within the respiratory system, and cause inflammatory reactions [40].

Regarding human health, microplastics have been observed in the blood, placenta, lung tissue, and gastrointestinal tract samples, and there is a worry that these compounds will have long-term physiological effects. Though the complete range of health hazards is still the subject of research, current data is strongly point to the fact that long-term exposures might indeed be among the factors of oxidative stress, inflammatory mechanisms, and possible genotoxic impacts [41].

## Future Perspectives and Analytical Challenges

Though advancement in the development of the microplastic detection technology is in progress, several problems with the analysis remain to be resolved to guarantee accuracy, comparability, and subsequent application in the real world. The lack of a consensus standard between sampling, pre-treatment, and analytical protocol is one of the greatest unresolved problems. Differences between reported concentrations are typical of various mesh sizes, digestion method, density separation media, and interpretation of the data. Without the universally accepted guidelines, comparisons between cross-studies are not consistent and present a challenge to reliable environmental monitoring systems [42].

Another challenge is in the process of identifying nanoplastics; they are the most biodegradable component, and probably more bioavailable in the environment. The existing available methods of analysis remain limited in the reliable detection of particles less than 1 mm. New high-resolution imaging methods and molecular analysis methods, including nano-FTIR, AF4-coupled detectors, and improved MS, are all on the verge of telling a more correct ID of nanoparticles, but it goes without saying that access to these techniques is very limited (costs, technological complexity, highly trained personnel) [43].

The other important constraint is the uncertification of the reference material (CRMs) of microplastics and their degradation products. Without the standardized reference particles, calibration and quantitation variation affect the inter-laboratory accuracy. In the process of preparing the CRMs, one should consider divergences in types of polymers, their degradation rates, and particle dimensions to make the method more robust [44].

The analytical development will definitely be geared towards various parameter multiparametric, hybrid systems with thermal mode coupled with spectral and chromatographic instruments within a single workflow. Such devices will enhance detection sensitivity, data throughput reduction, and minimize false positives will be minimized. Besides, it will be ideal to combine it with AI, automated imaging, and machine learning, which will, in turn, optimize pattern detection, quantitative analysis, and real-time monitoring at macro-levels.

Ultimately, it is possible to expect the regulatory regimes to expand in line with the rising evidence regarding health and ecological risks. Proper and comprehensive monitoring of microplastics and their degradation products would require the formulation of internationally harmonized standards, empowerment of testing facilities to do so, and the investment in new technology [45].

## Conclusion

Microplastics and their degradants have come to be the complex, multi-dimensional pollutants that require sophisticated methods of analysis to fully understand their fate, dynamics in environment/biology and long-term effects. Although the invention of spectroscopic, microscopic, chromatographic and thermal methods has yielded significant advancements in bridging these gaps, it is nonetheless large in specific areas on the detection of nano-sized particles, quantification of degradation byproducts as well as standardisation between laboratories. The different types of measurements have their own strengths and weaknesses that restrict the resolution of particle size, interference of the matrices, cost, and the access.

The further evolution of chemical characterization by hybridization with physical instruments is a potential trend towards the creation of more comprehensive data sets. The availability of promising new technologies such as nano-FTIR, AF4, machine learning-assisted image analysis, and high-resolution mass spectrometry (among others) is a clear indication that they can be further developed, although their wider application will require their affordability and methodological harmonization. Besides that, environmental and biological responses to microplastics, which include adsorption of contaminants, trans-cellular migration and release of toxic chemicals in the particles are posing an urgent need to develop improved methods of detecting the particles and risk-assessment protocols.

It will be necessary in the future to establish some universal standards concerning how the sample is collected, analysed, and pretreated so as to encourage reproducibility, comparability and reliability of findings across international research projects. The access to certified reference materials must be encouraged and multidisciplinary cooperation (environmental science, analytical chemistry, toxicology, computational tools) must be implemented in an attempt to accelerate the progress in this emerging branch of study. Lastly, to develop good regulations and mitigation measures along with sustainable reactions that would conserve ecosystems and human health as well.

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