


Overview of sugar distillery wastewater characteristics and the potential of nanomaterial adsorbents for treatment in Sindh, Pakistan

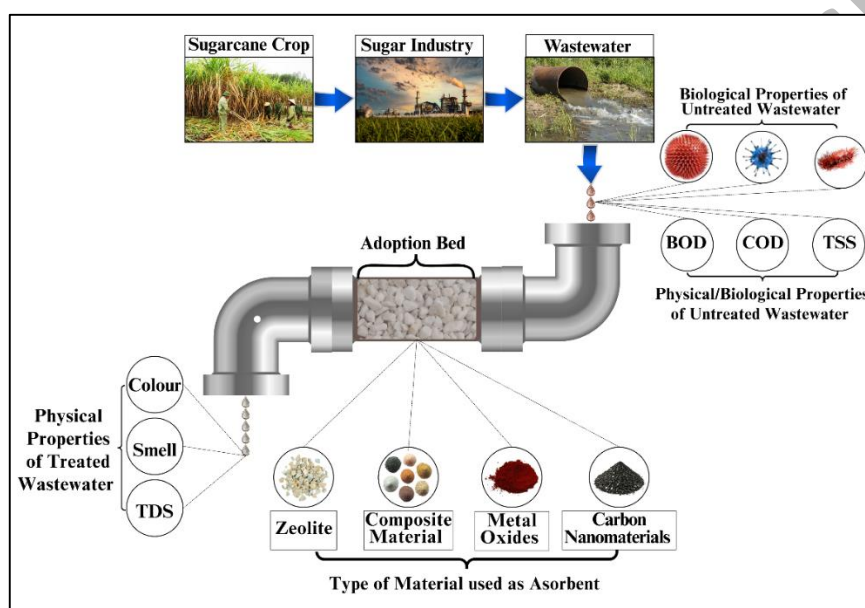
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ABSTRACT

Distilleries produce substantial amounts of wastewater containing high concentrations of organic and inorganic pollutants, leading to serious environmental concerns. In Sindh, Pakistan, water resources are at risk owing to the inappropriate disposal of industrial distillery effluents, contaminating land resources, water bodies, and groundwater. This has led to a notable increase in skin diseases and other health problems in Sindh, Pakistan. Therefore, effective and sustainable treatment strategies are required. We comprehensively reviewed the characteristics of sugar distillery wastewater from the Sindh region and highlighted its chemical composition, toxicity, and environmental impact. Conventional treatment methods often fail to achieve complete pollutant removal, necessitating the development of advanced alternatives. Nanomaterials, including nanoparticles, nanocomposites, and Nano-catalysts, have emerged as promising alternatives owing to their high surface areas, superior adsorption capacities, and reusability. This analysis examined a range of nanomaterials, such as metal oxides, carbon-based materials, and bio-inspired nanoparticles, and evaluated their potential for treating wastewater locally. This study aims to bridge the knowledge gaps and promote sustainable approaches for mitigating water pollution in the Sindh sugar distillery industry.

1. Introduction

Pakistan is an agricultural country and the production of agricultural commodities plays a vital role in its economy (Mahessar *et al.*, 2020). In recent years, distilleries have emerged as fast-emerging industries

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that produce ethanol and contribute immensely to Pakistan's economy due to the ubiquity of alcohol in other sectors, such as cosmetics, pharmaceuticals, and food (Lachenmeier *et al.* 2008; Dao *et al.* 2011). It is also used as a biofuel and is shared in the marketplace as an export product (Khan *et al.* 2007). Sugar crops provide almost 61% of the

ethanol production worldwide (Berg *et al.* 2004). In Sindh, Pakistan, most distilleries collaborate with sugar mills and use molasses from cane sugar manufacturing as a feed for alcohol production (Arshad *et al.* 2019). Currently, there are 21 distilleries in Pakistan with a production capacity of 240–270 liters per ton of molasses (Rashid and Altaf, 2008). Distillery is considered a highly contaminated industry because ethanol fermentation generates significant quantities of high-strength liquid effluents (Sultan *et al.* 2024; Siles *et al.* 2011). Alcohol-based sectors require a large amount of raw water, which produces a large amount of wastewater (Kharayat *et al.* 2012). The production of effluent streams from distilleries is termed sugarcane molasses wastewater (SCMW). High concentrations of contaminants, such as inorganic components such as sulfide and chloride, as well as volatile organic compounds (VOCs) such as ethanol and methanol, are characteristics of this type of wastewater. It typically exhibits a high chemical oxygen demand (COD) ranging from 80,000 to 160,000 mg/L, acidic pH, dark brown coloration, and a strong, unpleasant odor (Mikucka and Zielińska, 2020). The disposal of distillery wastewater without adequate treatment deteriorates the quality of the soil, water bodies, and, ultimately, groundwater, and the safe disposal of these effluents has become a growing concern as they negatively affect ecosystems and the health of residents (Ratna, Rastogi and Kumar, 2021).

Numerous treatment methods have been devised to control and degrade distillery wastewater toxicity, dark color, and high organic loads. These methods fall into three main categories: advanced oxidation processes (AOPs) (Fenton oxidation, ozonation, and photocatalysis), physicochemical (membrane filtration, coagulation and flocculation, and adsorption), and biological (aerobic, anaerobic, and fungal). Adsorption is an economically feasible, cost-effective, simple, and conventional process for producing high-quality products. Moreover, adsorption demonstrates high efficacy for the removal of a broad spectrum of contaminants, including both organic and inorganic compounds, underscoring its versatility as a wastewater remediation strategy (De Gisi *et al.*, 2016). Nanomaterials are well known and are frequently suggested for use as adsorbents because of their high surface area, which provides them with effective energy sites and efficient adsorption capability. Adsorbents with nano-scale structures are favored over prosaic materials because of their diverse nature, affordability, and environmental friendliness.

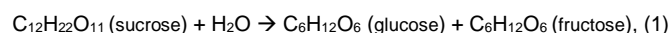
The objective of this review is to present a comprehensive analysis of the production, physicochemical characteristics, and environmental impacts of sugar distillery wastewater, with a focus on the industrial and ecological contexts of Sindh, Pakistan. To examine and evaluate the potential of adsorbents for the effective treatment of wastewater from sugar distilleries, and to offer a summary of the many nanomaterial adsorbents used in wastewater treatment, including information on their adsorption processes, types of nanomaterials used, treatment effectiveness, ideal operating parameters, and environmental sustainability. This study intends to critically evaluate distillery wastewater management within Sindh's socioeconomic and industrial setting by combining knowledge from the existing literature and contrasting regional treatment methods with international developments. It looks for gaps in local research activities, identifies limitations in current treatment technologies, and suggests possible avenues for further research and technological advancement. The ultimate goal of this review is to aid in the creation of ecologically friendly, scalable, and reasonably priced nanotechnology-based wastewater treatment solutions tailored to the needs of Sindh's sugar distillery industry, as well as other similar agro-industrial regions.

1.1. Background of sugar distillery industry and its wastewater

Ethanol (C₂H₅OH) from distilleries is in demand as a renewable fuel worldwide (Khan *et al.* 2021). In Pakistan, the production of ethanol is increasing in large numbers (Khan *et al.* 2019). Alcohol can be produced from various feedstock. Starch-based (corn, wheat, rice, and barley), sugar-based (sugarcane, beet molasses, and cane juice), and cellulosic (crop residues, sugarcane bagasse, wood, and municipal solid waste) materials (Rutz and Janseen 2007). The feedstock was selected based on its cost, availability, carbohydrate content, and conversion efficiency into alcohol (Robak and Balcerek 2018).

Ethyl alcohol production in distilleries is based on four main processes: feed preparation, fermentation, distillation, and packaging (Fig.1). Initially, blackstrap molasses was diluted with water in proportion to attain 10-15% of sugar content in the solution. The diluted solution was then added to the fermenter, and the 5% active yeast culture (*Saccharomyces cerevisiae*) (Satyawali and Balakrishnan, 2008) was also fed to the column. The fermentation mixture was then treated using distillation columns. The ethanol obtained (95%) from fractional distillation is called rectified spirit, and the bottom product is a

brownish liquid known as distillery stillage. This mixture of ethanol and water was further heated and refluxed for several hours to obtain the absolute alcohol (99%) (Senthamil Selvi, 2013). The probable reactions for ethanol production are given in Eq. 1–2.



Various by-products and pollutants are generated during these processes. Wastewater, as a potential pollutant in distilleries, is generated from different sources such as cooling water, column washing, and boilers. Unfortunately, waste streams from distilleries are acidic, dark brown recalcitrant compounds with high biological oxygen demand (BOD), chemical oxygen demand (COD), and total dissolved solids (Shinde *et al.*, 2020), as summarized in Table 1. It also contains various salts, endocrine-disrupting chemicals (EDCS), and toxic heavy metals (Tripathi *et al.*, 2021). The large quantity of discharged effluents is a serious threat to terrestrial ecosystems.

Table. 1. Characteristics parameters of sugar Distillery wastewater

Parameters	Values
Volume (L/L alcohol)	10-15 l
pH	3.8-4.4
Temperature	Very high
Colour	Blackish Brown
BOD	40,000-60,000 mg/L
COD	80,000-100,000 mg/L
Total solids	90,000-120,000 mg/L
Total dissolved solids	30,000-40,000 mg/L
Total volatile solids	60,000-70,000 mg/L
Sulphates	4000-8000 mg/L
Chlorides	5000-6000 mg/L

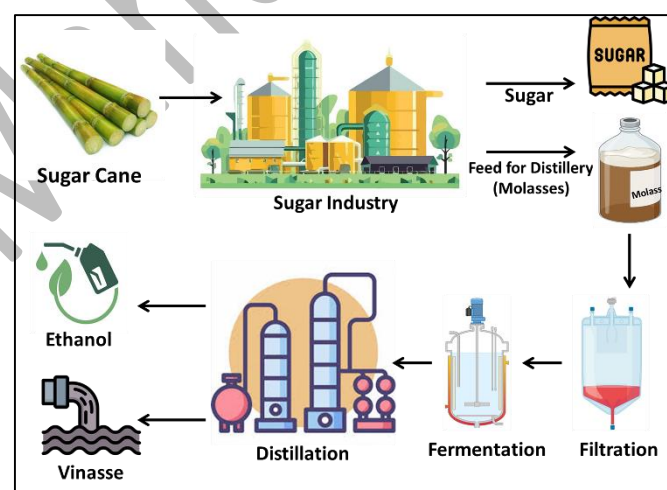


Fig. 1. Process description of sugar distillery industry and its wastewater.

1.2. Overview of sugar distillery wastewater, its characteristics & effects in Sindh, Pakistan

Wastewater generation from distilleries and fermentation industries is a major concern because of water scarcity and the huge volumes of wastewater management. The effluent generated from distilleries, also termed spent wash, is approximately 10-15 times the amount of alcohol produced per liter (Sankaran *et al.*, 2014). The physicochemical properties of distillery stillage differ based on the raw materials used and the operational processes involved in alcohol production, resulting in wastewater characteristics that vary among distilleries (Mohana, Acharya, and Madamwar, 2009). Wastewater contains high chemical oxygen demand (COD), biological oxygen demand (BOD), dissolved organic nitrogen, phenolics, total dissolved solids, phosphates, and sulfates because of the high load of organic compounds such as proteins, waxes, polysaccharides, and heavy metals (Chaudhari, Mishra and Chand, 2007). The dark brown color of the effluent stream is due to the presence of condensation products of amino acids, proteins, and carbohydrates, termed melanoidin (Chandra, Bharagava, and Rai, 2008). Melanoidin constitutes approximately 2% of distillery wastewater (Kaushik *et al.*, 2018). The obnoxious smell in the spent wash is due to the presence of sulfur compounds such as indole and skatole (Bhardwaj *et al.*, 2019). Some Distilleries in Sindh, Pakistan, treat spent wash through anaerobic digestion (Salahuddin Panhwar *et al.*

al., 2019). Although COD and BOD are removed to a great extent through anaerobic digesters, the color remains because the colored compounds are non-biodegradable (Malik *et al.*, 2018). Melanoidin also possesses antioxidant properties that are toxic to microorganisms present in conventional wastewater treatment (Pant and Adholeya, 2007). Distillery wastewater also contains endocrine-disrupting chemicals (EDCS), which lead to hormonal irregularities that trigger different metabolic and physiological disorders, ultimately compromising reproductive fitness in both humans and animals (Chowdhary, Raj and Bharagava, 2018), (Gonsioroski, Mourikes, and Flaws, 2020). A large volume of dark-colored wastewater pollutes both water bodies and landfills. They hinder sunlight penetration, disrupt photosynthetic activity, and lead to the depletion of dissolved oxygen, posing a serious threat to aquatic life. When exposed to vinasse, *Oreochromis niloticus* (tilapia) exhibited dose-dependent acute toxicity, distinguished by cytoplasmic degeneration and a substantial decrease in hepatic polysaccharide accumulation, indicating a major metabolic disturbance (Urbano *et al.*, 2014). Additionally, their high organic loads

can cause eutrophication when discharged into water bodies (FitzGibbon *et al.*, 1998). Distillery wastewater can adversely affect landfills in Pakistan (Bano and Arshad, 2017) by inhibiting seed germination due to Mn deficiency and soil alkalinity (Fuess and Garcia, 2014). Prolonged irrigation with post-methane distillery effluent (PMDE) increases soil fertility by increasing phosphorus, potassium, total organic carbon (TOC), total Kjeldahl nitrogen (TKN), and enzymatic activity. However, it also causes Na to accumulate in the soil, which could be harmful to the long-term viability of agriculture (kaushik *et al.* 2005). The amounts of Fe, Zn, Ni, Cd, and Cr in *Spinacia oleracea* (spinach) plants and the surrounding soil have been reported to increase when distillery effluents, particularly vinasse, are used for irrigation. This study suggests that prolonged application of vinasse poses a significant risk of heavy metal accumulation in crops, thereby raising concerns regarding the potential adverse effects on food safety and human health (Pathak *et al.*, 2014). The textual description was supplemented with a graphic overview of this pathway (Fig. 2).

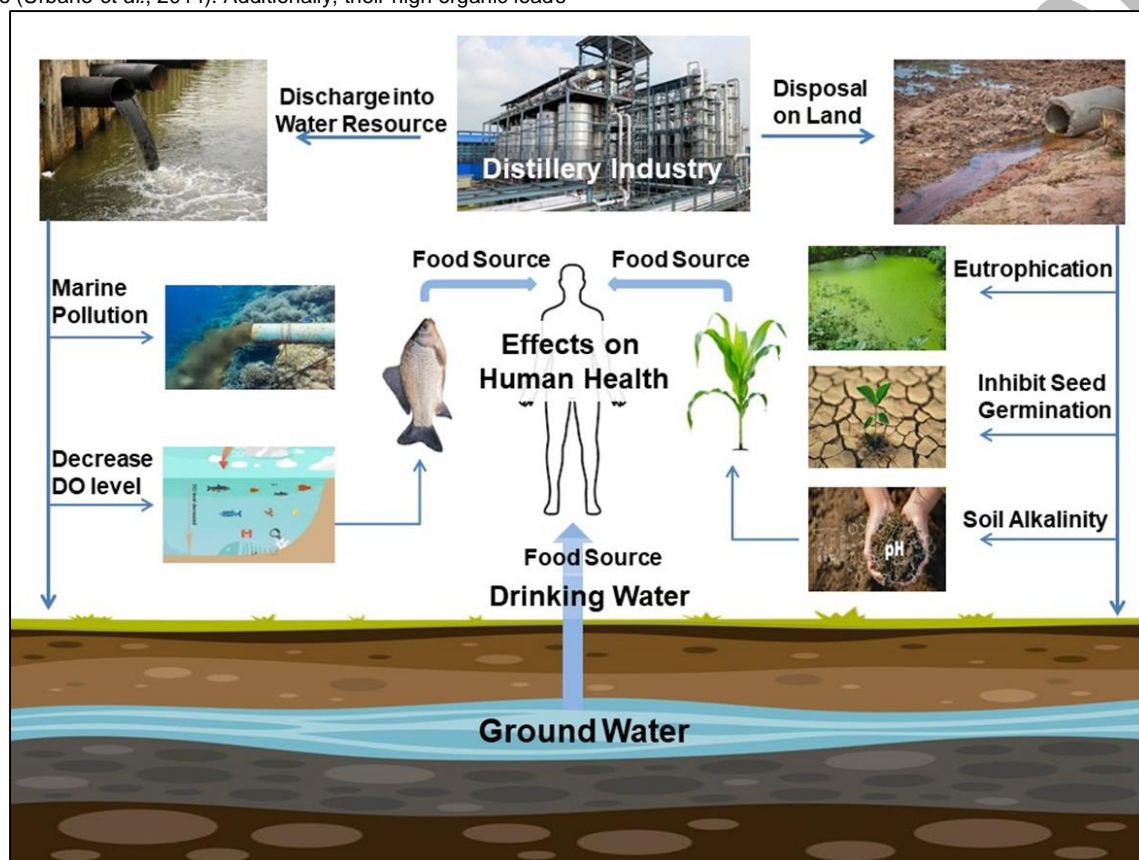


Fig. 2. Distillery wastewater, characteristics and effects.

In Pakistan, untreated wastewater from distilleries is often dumped into rivers, streams, and other waterways, severely damaging the groundwater. Studies have shown that the quality of groundwater has decreased in places where sugar manufacturers release wastewater with high concentrations of total dissolved solids (TDS) and other pollutants. This contamination poses a serious risk to local communities due to skin disorders and waterborne diseases (Qureshi *et al.*, 2015). Additionally, research indicates that groundwater pollution from industrial activities in Pakistan affects drinking water quality and puts human health at risk (Soomro *et al.* 2011). Therefore, the removal of color and a high organic load from distillery wastewater is necessary for a better environment and sustainable development (Chowdhary *et al.*, 2017). The inability of the current practices and concentration achieved in the laboratory is quite low in comparison to the guidelines put forth by environmental standards, as well as the first international guidelines for water reuse in agriculture (Directive EU *et al.* 2020). In an effort to satisfy the stringent government pollution control regulations, industries are being compelled to implement remediation technologies that are both economically viable and environmentally favorable in order to achieve more sustainable and efficient outcomes. (Faruqee and Kemal, 1996).

1.3. Importance of color removal and enhancement of physical and biological properties of sugar distillery wastewater in Sindh

The disappearance of color and enhancement of physical and biological qualities are of foremost importance and cannot be surpassed (Shahnawaz *et al.*, 2020; Collivignarelli *et al.*, 2019). The vision of an environmental engineer is referred to as visual engineering. The removal of color is of great importance, as it refines water. Color significantly contributes to aesthetic excellence and blemishes water quality for application in irrigation and effluent discharge into local water bodies (Mehmood, Batool, and Qazi, 2013). The deep coloration of distillery wastewater is caused by organic compounds such as phenolics, melanoidins, furfurals, and caramels (Miranda *et al.*, 1996). Melanoidin is the main contributor of the dark brown pigment in wastewater (Arimi *et al.*, 2015). The dark color of wastewater impedes photosynthesis, leading to a reduction in the levels of dissolved oxygen in water (Tariq *et al.* 2022; Tirpathi *et al.* 2021). With regard to aquatic life, a decline in dissolved oxygen supports anaerobic organisms in aerobic digesters, which in turn produce hydrogen sulfide (Muller *et al.* 2022). Hydrogen sulfide can deteriorate the canal structure (Zhang *et al.*, 2023). Consumption of untreated wastewater can lead to skin allergic effects and possibly other waterborne diseases in the concerned population (Afzal *et al.*, 2018). Distillery wastewater is naturally turbid (Bezueh and Kebede 2015). Turbidity is caused by the presence of soluble organic matter and suspended matter (Momeni *et al.* 2018; Kumar *et al.* 2014). Conversely, an increase in turbidity reduces the amount of solar radiation (Shedayi *et al.* 2016). Improvements in the physical properties will not only refine the

color but can also elevate the organic configuration in wastewater, making it useful for further applications (Zielińska, Bułkowska, and Mikucka, 2021).

A reduction in the turbidity of wastewater will also contribute to a reduction in the chemical oxygen demand (COD), as highly turbid wastewater allows for higher COD values during analysis (Nguyen, Ward, and Lewis, 2014). It is envisaged that the backbone of any treatment facility is its treated effluent, which experiences improvement not only in terms of organic and inorganic configuration, but also in other general parameters (Lako & Çomo 2024). The agricultural sector is largely dependent on water from rivers and natural rain-fed canal streams, which, on their way, capture and flow through the open drains of big cities. The use of effluents in sewer drains with high total suspended solids (TSS) and TDS values imperils irrigated cultivation, fruit trees, vegetation, and traditional livestock (Awan et al., 2012). Wastewater treatment technologies should not only be environmentally friendly, but also cost-effective, as environmental measures can burden the socio-economy of the country (Khan et al. 2022). The drainage of undiluted wastewater can also impose a percentage of the total country water resources to dilute and sustain existing natural water flow in streams and rivers. A grassroots-level approach is required to tackle this issue (Martí, Riera, and Sabater, 2009). Therefore, management measures require effective design and implementation of advanced wastewater treatment systems (Zhou and Smith, 2002). The subject wastewater should comply with the effluent standards for two reasons: one is self-imposed, and the second is that in the best national scenario, the same water could be utilized for irrigation techniques that can increase the number of crops and land with the same output (Ali, Pervez and Khan, 2020). Proper management of the effluent stream enhances its physical and biological properties, and it can be utilized as a soil fertilizer (Cerri et al., 2020). It is anticipated that effluents from advanced treatment technologies can be used for the growth of hardwood trees, aquarium fish culture, environmental aesthetics, landscape water bodies, golf courses, surface gardening, and dust suppression in mining and construction. Ultimately, the paramount objective of environmentalists is to maintain a healthy environment (Khalid et al. 2018; Hanjra et al. 2012).

1.4. Potential of nanomaterial adsorbents for wastewater treatment in the context of Sindh

Nanomaterials are tiny artificial structures that are nanometers in size. They are more efficient than conventional materials and can be used to remove organic and inorganic compounds from wastewater (Younas et al., 2021). Nanomaterials exhibit exceptional properties and show promising results as adsorbents for contaminant removal from wastewater. These adsorbents are capable of treating a variety of pollutants, such as organic pollutants (e.g., color, medications, and insecticides) and heavy metals (e.g., lead and mercury). Innovative adsorbent research also encompasses emerging contaminants such as per- and poly-fluoroalkyl substances (PFASs), microplastics, and endocrine-disrupting chemicals (Akhtar, Ali, and Zaman, 2024). The structural characteristics of nanomaterials, such as their high surface area and reactivity, enhance their effectiveness in removing heavy metals (Fu and Wang, 2011). Nanotechnology has been explored as a transformative approach to wastewater treatment. nanomaterials are categorized into three groups: nano-adsorbents, nanomembranes, and nano-catalysts (Anjum et al., 2019). Various types of nanomaterials, including metals, metal oxides, carbonaceous materials, and bio-based nanomaterials, have demonstrated significant color-removal efficiencies (Umar et al., 2023; Saleem, 2023). In particular, magnetic-based nanomaterials can remove more contaminants in less time and can be reused (Asghar et al., 2024; Karnwal and Malik, 2024). Surface-modified magnetic nanoparticles with tridentate ligands effectively remove heavy metals. The Pb²⁺ and Cd²⁺ adsorption capacities were 93.5 mg/g and 83.5 mg/g respectively. (Masjedi, Askarizadeh and Baniyaghoob, 2020). Sarwar et al. (2024) examined the potential of biopolymeric nanocomposites for wastewater remediation by focusing on their production methods, properties, and applications. Despite the potential of nanomaterials, there are challenges such as environmental toxicity, cost, and operational issues that need to be addressed on a large scale (Muzammil et al., 2023). Future research should focus on developing improved nanomaterials and composite materials with high adsorption capacity, selectivity, and durability to provide durable and recyclable solutions for the treatment of sugar distillery wastewater in Sindh (Shahzad et al., 2025; Saleem et al., 2020).

2. Synthesis, type and characterization of nanomaterial adsorbents

2.1. Synthesis of nanomaterial adsorbents

The synthesis of nanomaterial adsorbents should be developed to achieve a green, time-saving, scalable, and simple approach to prepare nano adsorbents with high surface areas and good stabilities for wide applications (luo et al. 2021). Nanomaterial adsorbents are commonly synthesized by chemical, physical, and biological processes and are widely used for the fabrication of nanomaterials (Ullah et al. 2019) (ref to Fig. 3).

They (the methods of synthesis of nanomaterials) can be broadly classified into two scientific approaches: (i) top-down and (ii) bottom-up. The breakdown of bulk materials into their nanosized structures is equivalent to the top-down approach. In contrast to the bottom-up approach, nanostructures are constructed from atomic or molecular units (saleh et al. 2021).

Top-down synthesis is mostly carried out using physical methods, such as electron beam lithography (Avoyan, Rupprechter, and Eppler 2000), etching (Du et al. 2014), high-energy ball milling (Amusat et al. 2021), inert gas condensation (Turker et al. 2004), and sputtering (Verma et al. 2017). These techniques are highly effective for producing well-ordered structures with macroscopic crossovers; however, the surface morphology may not be ideal. However, bottom-up synthesis is mostly done chemically through sol-gel synthesis (Tabesh, Davar, and Loghman-Estarki, 2018), hydrothermal synthesis (Ilu et al. 2014), H₂O₂ synthesis (Li et al. 2012), CVD (alyan et al. 2018), solvothermal synthesis (Chella et al. 2014). One of the most popular methods for synthesizing nanomaterials, such as metal/metal oxide nanoparticles and carbon-based nanomaterials, is the use of these methods because they can form uniform, small particles and include both inorganic and organic components (Patricio, López, and Thirumuruganandham, 2023).

Following the bottom-up pathway, biological methods use microorganisms, plants, and enzymes for the synthesis of nanomaterials, providing the advantages of biological friendliness, low cost, and high biosafety (Jamshed et al., 2023). In particular, for synthesizing eco-friendly adsorbents, such as biosynthesized metal nanoparticles and plant-based carbon nanomaterials, these methods are convenient and an ideal choice for synthesis in areas with resource constraints (Rafatullah et al. 2010). Biological synthesis using naturally occurring substances is safer and more sustainable than physical and chemical methods based on secondary chemicals, often toxic chemicals, and expensive reagents (Ijaz et al. 2020). With the significance and environmental problems of the Sindh province, biosynthesis is expected to be a promising arena for future research in material synthesis and environmental purification. Local organic resources and biomass waste are emphasized as proper advancements in green nanotechnology to synthesize wastewater adsorbents suitable for local applications (Akhtar, Sarfraz, and Ahmad, 2024). Effective remediation requires novel synthetic strategies appropriate for the climate of Sindh. Ikram et al. (2023) demonstrated the feasibility of conventional synthesis methods for producing nanomaterials suitable for Sindh. The conversion of biomass waste to nanomaterials is advantageous from both environmental and economic perspectives, particularly for the development of adsorbents such as carbon nanomaterials and nanocomposites for industrial applications (Mubarak et al. 2021; Tariq, Yahaya, and Sajid, 2024).

2.2. Types of nanomaterial used for distillery wastewater remediation

Nanostructured adsorbents, owing to their remarkably high surface areas compared to conventional materials, have become relatively common because of their faster adsorption rates and efficiencies. These top-down, bottom-up, and green synthesis nanomaterials are broadly categorized as carbon-based, metal/metal oxide, oxide-based, composite, and zeolite-based nanomaterials. Furthermore, some of these nanomaterials, which have distinctive functions, can be employed for the detoxification of distillery wastewater, which is differentiated as carbon-based (Nure et al., 2017), metal/metal hybrid nanoparticles (Manikandan et al., 2022), oxides, composite materials (El-Dib et al., 2016) and nanoscale zeolite materials (Apollo et al., 2019). Consequently, these nanomaterials can be effectively applied to degrade the majority of pollutants residing in distillery effluents, that is, color, COD, BOD, organic phenols, and heavy metals.

For effective waste management, the focus is on locally obtained carbon-based and metal/metal oxide nanoparticles in the Sindh Province (Ahmad et al., 2020). Activated carbon, carbon nanotubes, and graphene are potential candidates for remediating distillery wastewater because of their high surface area and high adsorption capacity. Specifically, adsorbents such as activated carbon (AC) are frequently employed owing to their relatively high surface area and porosity, as well as their strong affinity for organic compounds.

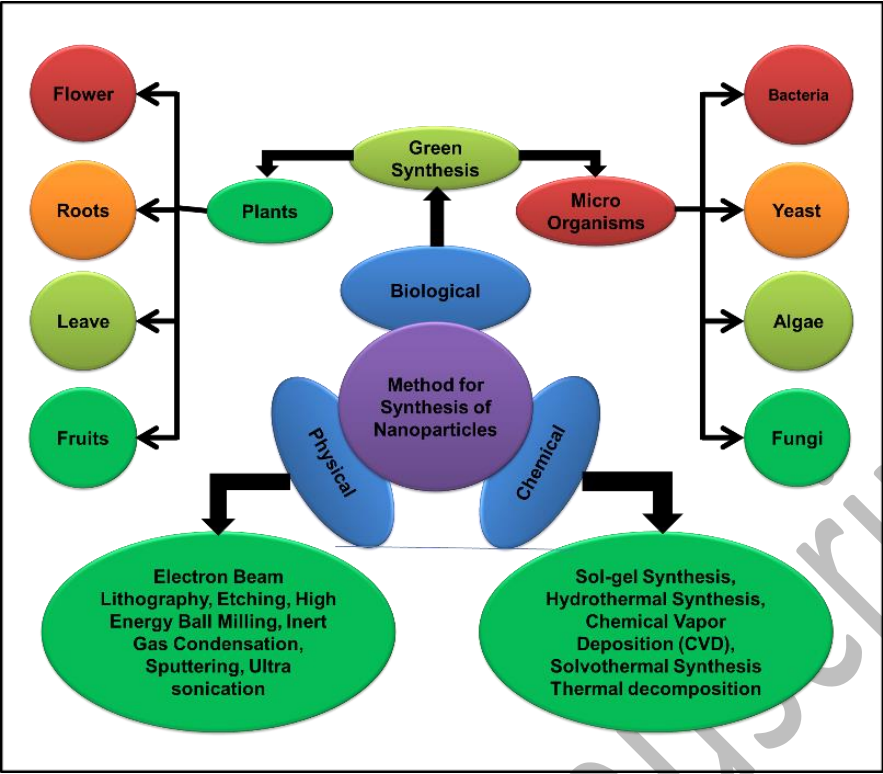


Fig. 3. Synthesis methods of nanomaterial adsorbents.

AC can be synthesized from abundant and economical materials such as agricultural residues (Almadani *et al.*, 2023), AC can be synthesized and has been found to efficiently adsorb pollutants such as organic phenols, COD, and color. Inorganic nanomaterials, such as metal or metal oxide-based nanomaterials, are widely used for wastewater treatment because of their catalytic properties and factors for specific pollutant removal (Naseem & Durrani, 2021). Therefore, detoxification of distillery effluents using magnetic nanoparticles (Fe₃O₄) has been demonstrated through the use of heavy metals as well as the removal of organic compounds and COD (Mahmoudabadi, Rashidi, and Maklavany, 2022). Propagation studies have also been conducted on distillery wastewaters. Contaminants have been removed using nanomembranes, nanofibers, and other nanomaterial-based membranes (Nataraj, Hosamani, and Aminabhavi, 2006).

Nanocomposites (NCs) are innovative materials formed by the combination of two or more different components, namely, the matrix

and reinforcing phase, to produce a comprehensive set of properties superior to the properties of the individual constituents. These materials are also used to enhance the adsorption capacity and stability of the process for the removal of complex pollutants, such as aromatic compounds, COD, and color from distillery wastewater treatment (Ashraf *et al.*, 2024; Nadeem *et al.*, 2021).

2.3 Characterization techniques of nanomaterial adsorbents

Characterization of nanomaterials is crucial for the development of repeatable synthesis techniques. Characterization involves examining the structure, composition, and range of electrical, magnetic, chemical, and physical characteristics. Numerous methods are available for characterizing nanomaterials (Kumar *et al.* 2019). The characterization techniques can be classified into three categories (Tables 2, 3, and 4): chemical, physical, and thermal characterizations. (Pellenz *et al.* 2023).

Table 2. Chemical characterization techniques for nanomaterials.

	Technique	Purpose	Key outputs	References
Chemical characterization	X-Ray Photoelectron Spectroscopy (XPS)	Determines surface elemental composition, oxidation states, and chemical bonding.	Binding energies, oxidation states.	Steve and Donley 2020
	Fourier-Transform Infrared Spectroscopy (FTIR)	Identifies functional groups and surface chemical bonding.	IR absorption spectra.	Smith, 2011
	Raman Spectroscopy	Probes molecular vibrations and chemical structure.	Raman shifts, peak intensity.	Ferrari and Basko 2006
	Inductively Coupled Plasma Mass Spectrometry (ICP-MS)	Quantifies trace elements with high sensitivity.	Concentration of metal ions.	Wu et al, 2009
	UV-Visible Spectroscopy (UV-Vis)	Analyzes adsorption properties and optical behavior.	Absorption peaks, band gap energy.	Akash and Rehman, 2020
	Nuclear Magnetic Resonance (NMR)	Examines molecular structure and chemical interactions.	Chemical shifts, bonding environments.	Levitt, 2008
	X-Ray Fluorescence (XRF)	Identifies and quantifies bulk elemental composition.	Elemental composition.	Jenkins, 1999

3. Adsorption mechanisms and performance

3.1. Mechanism of removal of existing pollutants by nanomaterials

Nanomaterials are widely used for the removal of pollutants from sugar distillery wastewater using various adsorption, catalytic degradation, ion exchange, and membrane-based filtration mechanisms. The

interactions of nanomaterials range from unique physicochemical properties, such as large surface area, enhanced porosity, high surface reactivity, and the presence of active functional groups, to color compounds, COD, BOD, organic phenols, and heavy metals (Dhila, Bhapkar and Shekhar Bhamé, 2025). Currently, pollutants are physically or chemically bound to the surface of nano adsorbents such

as activated carbon, graphene oxide, and carbon nanotubes (R.Hari Krishna *et al.*, 2023). In photocatalytic degradation and redox reactions, metal oxide nanoparticles, such as TiO₂, ZnO, and Fe₃O₄ play a significant role in removing complex organic molecules and reducing toxicity levels (Li *et al.*, 2020). Also, magnetic nanoparticles make it easy to separate and retrieve the nanomaterials (Soylak, Ozalp, and

Uzcan, 2021). Nanocomposites and hybrid materials often inherit multiple mechanisms where the combinations, in turn, are synergistic to increase the overall treatment performance (Ribeiro, Dias, and Zille, 2022). This multifunctionality makes nanomaterials a highly suitable and sustainable option for the treatment of distillery effluent wastewater at low dosages in Sindh, Pakistan.

Table 3. Physical characterization techniques for nanomaterials.

Physical characterization	Technique	Purpose	Key outputs	References
	Scanning Electron Microscopy (SEM)	Studies surface morphology, particle size, and distribution.	High-resolution surface images.	Goldstein <i>et al.</i> , 2003
	Transmission Electron Microscopy (TEM)	Probes internal structure, crystallinity, and nanoparticle size.	Atomic-scale images, lattice fringes.	Zuo and spence 2017
	Atomic Force Microscopy (AFM)	Measures surface roughness, topography, and mechanical properties.	3D surface profiles, roughness values.	Voigtländer 2019
	X-Ray Diffraction (XRD)	Determines crystalline structure, phase purity, and grain size.	Diffraction patterns, lattice constants.	Cullity and Stock, 2001
	Dynamic Light Scattering (DLS)	Measures hydrodynamic diameter and size distribution in suspensions.	Particle size distribution.	Stetefeld, McKenna and patel, 2016
	Brunauer-Emmett-Teller (BET) Analysis	Measures surface area, pore size, and pore volume of porous materials.	Specific surface area, pore distribution.	Brunauer, Emmett and teller 1938
	Zeta Potential Measurement	Evaluates surface charge and colloidal stability in aqueous solutions.	Zeta potential values.	Clogston and Patri 2010
	Thermogravimetric Analysis (TGA)	Analyzes thermal stability and adsorbent decomposition behavior.	Weight loss vs. temperature.	Brown, 2004
	Surface Area Analyzer (SSA)	Measures specific surface area and pore structure.	BET surface area, pore volume, pore diameter.	Sing, 1982
Thermal characterization	Contact Angle Measurement	Measures surface wettability and hydrophilicity / hydrophobicity.	Contact angle values.	Kwok and Neumann, 1999

Table 4. Thermal characterization techniques for nanomaterials.

Thermal characterization	Technique	Purpose	Key outputs	References
	Thermogravimetric analysis (TGA)	Evaluates thermal stability, decomposition behavior, and residual mass.	Weight loss vs. temperature.	Rami <i>et al.</i> , 2021
	Differential scanning calorimetry (DSC)	Measures heat flow associated with phase transitions (e.g., melting, crystallization).	Glass transition temperature, melting point.	Koshy, Subramanian, and Thomas 2017
	Differential thermal analysis (DTA)	Identifies endothermic or exothermic reactions during heating.	Temperature difference between sample and reference.	Mansfield, 2015
	Thermomechanical analysis (TMA)	Measures dimensional changes in response to temperature.	Thermal expansion coefficients.	James, 2017
	Dynamic mechanical analysis (DMA)	Studies viscoelastic properties of nanomaterials under thermal stress.	Storage modulus, loss modulus, damping factor.	Bashir, 2021

3.2. Nanomaterial adsorption mechanisms

Adsorption is an essential process in wastewater treatment because it contributes significantly to the purification of pollutants (Rashid *et al.*, 2021). Adsorption is a mass transfer process in which a substance from the liquid phase is transferred onto a solid surface (Alaqrbeh, 2021). When a solute solution and a highly porous solid material come into contact, certain solute molecules are pulled to the solid by intermolecular forces, which leads to the solute molecules adhering to the solid's surface or accumulating (Sadegh *et al.* 2017). During adsorption, the attractive forces on the adsorbent surface outweigh those on the dissolved components of the liquid. The forces between the adsorbate and adsorbent can be physical or chemical (Bhavsar *et al.* 2021). In the former, physisorption involves weak forces, such as hydrogen bonding, van der Waals forces, and electrostatic interactions. It can purify pollutants through physical interactions between substrates, called adsorbents, and contaminants, called adsorbates (Zang and Xing, 2011). Electrostatic and hydrophobic interactions govern pollutant removal, damaging water quality and ecosystems (Mandal *et al.*2024). According to Xiong *et al.* (2024), amine-functionalized soy protein/GO aerogels have high affinity for compounds derived from sugars. The adsorption process is mainly driven by electrostatic (charge) interactions between the protonated amine groups and anionic sugar moieties, in addition to extensive hydrogen bond formation at the protein–graphene oxide interface. Nunes *et al.* (2015) used activated carbon to study melanoidin removal and found that π-π interactions and hydrogen bonding were the main adsorption mechanisms involved. Chemisorption, that is, chemical

adsorption, occurs as a result of electron transfer and chemical bonding (Agboola and Benson, 2021). Gonzalez-Valerio *et al.* (2024) demonstrated that melanoidin adsorption on silica gel functionalized with thiol moieties occurred predominantly via chemisorption. This study also showed that thiol groups on the adsorbent surface can form covalent bonds with melanoidin, specifically through interactions with pyrazinium compounds derived from the Maillard reaction. Multiple studies have demonstrated that functionalized adsorbents exhibit a robust and specific affinity for target organics.

Different adsorption mechanisms are illustrated in Fig. 4. To understand the adsorption mechanisms and boost the adsorption process, adsorption kinetics and isotherm models (Karimi, Tavakkoli Yaraki, and Karri, 2019). The adsorption isotherm is useful for representing the capacity of an adsorbent and describing the functional dependence of the capacity on the concentration of pollutants (Saleh, 2022). Adsorption kinetic studies demonstrated the rate of adsorption and identified the adsorption mechanism. There were three main steps in this process. The adsorbate is first transported to the outside of the adsorbent via surface coating (also known as "film diffusion"); second, the adsorbate diffuses into the adsorbent's pores (also known as "pore diffusion"); and third, the solute adsorbs on the interior surfaces enclosing the capillary and pore gaps. (Díaz, García and Ordóñez, 2024). The performance of the adsorption process depends on the nature of the adsorbent, its physical attributes, and the process parameters (Sen, 2023).

3.3. Factors affecting adsorption process efficiency

The factors affecting adsorbent performance are interconnected with the wastewater being treated (Satyam and Patra, 2024). The physical and chemical properties of the adsorbent, such as the adsorption number, nanoparticle size, surface area, functional groups, and mesoporous distribution, play significant roles (Badran *et al.*, 2023). Smaller nanoparticles have higher adsorption efficiency owing to their larger surface area (Wang and Shadman, 2012). Nanoparticles operationalized with attached oxidizing and reducing sugar molecules are more reactive and exhibit better adsorption capabilities (Wang *et al.*, 2025). Functionalizing adsorbent surfaces with different functional groups and composite materials provides charged and synergistic effects (Olawade *et al.* 2024). The number of accessible active sites for adsorption to take place increased with increasing adsorbent dosage, improving adsorption effectiveness. However, an increase beyond a certain point also results in the aggregation of nanoparticles and a reduction in the surface area and active sites, which diminishes the adsorption capacity (Singh *et al.* 2021). The chemical composition of nanoparticles also affects adsorption, influencing their surface characteristics and nature. The adsorption capacity increased with nanoparticles (Naseer, 2024).

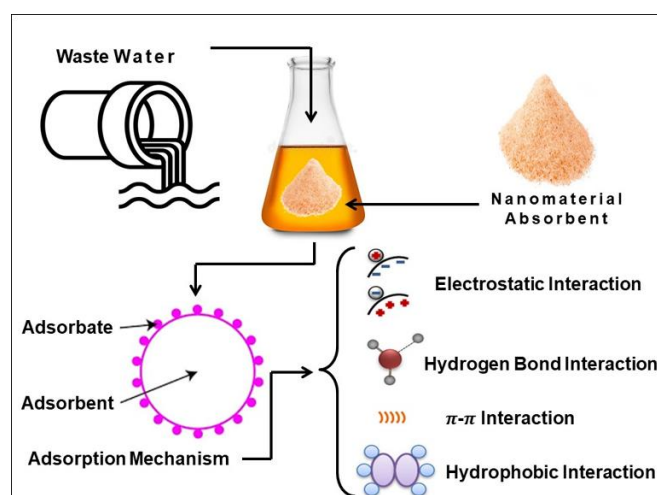


Fig. 4. Adsorption mechanism of nanomaterial adsorbents.

Another crucial factor that significantly influences the sorption capacity of wastewater is the pH (Wang *et al.* 2005). Variations in pH affect the degree of ionization, speciation of pollutants, and charge on the adsorbent surface. Furthermore, a change in pH disrupts the adsorption process by dissolving the functional groups on the active sites of the adsorbent surface. Consequently, the equilibrium and kinetics of the adsorption process are altered (Ahmed *et al.*, 2020). Fan *et al.* (2022) studied the effect of pH on the uptake of melanoidin and observed that PMPC has a better adsorption efficiency for melanoidin in the pH range of 5–9.

The result is explained by the electrostatic interaction between the negatively charged melanoidin and the very positively charged PMPC. Interestingly, beyond the pH of PMPC, the surface charge of the adsorbent reverses to a negative charge, leading to decreased adsorption owing to the electrostatic repulsion between the similarly charged species.

In addition, factors such as contact time, mass transfer resistance, initial/final concentrations, and concentration steps can influence adsorption kinetics (Raji *et al.*, 2023). Longer adsorption times result in increased chemical conversion and faster adsorption (Mariana *et al.*, 2021). Saturation and aggregation also impede solute transportation and negatively impact the mass transfer resistance (Dou *et al.*, 2022). High solute concentration steps are unfavorable because they lead to the saturation of nanoparticle sites, hindering solute removal (Rapo and Tank, 2021). (Rafiq and Rahimpour Soleymani 2019) investigated the effect of the initial solute concentration of the melanoidin solution from 600 mg-1 to 1300 mg-1.

It was observed that as melanoidin concentrations increased up to 18.02 g-1, the GONs adsorption capability of GONs increased dramatically before declining substantially. This trend is explained by the rapid initial adsorption on the available sites, followed by saturation of the surface. Temperature is a significant variable that affects the adsorption capacity. This process is endothermic when the adsorption capacity increases with temperature. The decline in the adsorption capacity with increasing temperature implies that adsorption is an exothermic process (Budnyak *et al.*, 2020). Li *et al.* (2020) discussed the effect of temperature on RP-Si-CA adsorbents. The findings

indicated that an increase in temperature increased the adsorption capacity of melanoidin, implying that the process was endothermic.

3.4 Removal efficiency of nanomaterial adsorbent in context of distillery wastewater

Ayub *et al.* (2014) reported the effective performance of activated carbon as an adsorbent. During 24 h of process time and at an optimum dosage of 10g/200 ml, the removal of COD, TDS, and DO at the levels of 93.33, 70.6, and 84.8 %, respectively, was observed. Sobande *et al.* (2024) studied the effect of orange peel carbon on melanoidin treatment using batch adsorption. They achieved 60.20 % removal at optimum conditions of 4 pH, 2.5g/100ml adsorbent dosage, and one h retention time. Reyes *et al.* (2017) studied melanoidin removal through batch experiments using different forms of carbon, and found that carbon multi-walled nanotubes achieved the highest removal adsorption capacity of 5.3 mg/g. Wang *et al.* (2024) examined the performance of polyethylene glycol cross-linked modified chitosan/halloysite nanotube composite aerogel microspheres (PCAM@HNTs) for melanoidin degradation, and the removal rate was 98.53% at pH 7, 303K and 0.4mg/ml adsorbent dose. Kiran *et al.* (2015) investigated the treatment of distillery spent wash using mixed matrix membranes (MMMs) incorporating graphene oxide (GO) flakes into polyethersulfone (PES). Maximum color removal of 54% was achieved for the distillery spent wash effluent. Srivastava *et al.* (2021) examined the zero-valent iron nanoparticle treatment efficiency of distillery wastewater, which was synthesized and characterized as a green approach. They recorded maximum reductions in color, COD, BOD, and TSS were 99.07%, 84.25%, 89.23%, and 86.5%, respectively. Kazemi *et al.* (2015) investigated the catalytic efficiency of CuO and MnO in the treatment of distillery wastewater in a tubular reactor under hydrothermal conditions. They found the COD removal efficiencies of CuO and MnO₂ were 74.4% and 75.1%, respectively. They also achieved 98.2% color removal using MnO₂ at 400°C. Otieno *et al.* (2017) investigated the photo-decolorization of vinnase melanoidin using a composite nanomaterial adsorbent comprising zinc oxide (ZnO) immobilized on titanium dioxide supported by activated carbon (TiO₂-ZnO/AC) in a batch reactor. The study revealed that the hybrid material (TiO₂-ZnO/AC) achieved up to 86% decolorization. Wang *et al.* 2022 studied the removal of melanoidin from sugarcane juice using a bagasse-based biochar composite with hydroxyapatite and obtained an excellent adsorption capacity of 313.33 mg/g. Navgire *et al.* (2012) have reported 70% of color removal from molasses solution using nanocrystalline composite material MoO₃-TiO₂ by photodegradation within 120 min time duration. Donyagard *et al.* (2017) examined melanoidin removal using a magnetic carbon nanocomposite as an adsorbent. They reported 90% removal in simulated aqueous solution and 80.76% in real wastewater at a concentration of 40 ppm. Onyango *et al.* (2011) investigated the sorption of melanoidin on surfactant-modified zeolites and reported an adsorption capacity of 1157.0 mg/g at a temperature of 45 °C. Tang *et al.* 2023, fabricated a hyperbranched polyethyleneimine-functionalized chitosan aerogel (HPCA) for melanoidin removal. HPCA demonstrated an efficient adsorption capacity of 868.36 mg/g.

4. Comparative analysis: Sindh v/s Global practices

Effluents from the distillery industry pose health risks owing to the lack of appropriate waste-treatment facilities. In recent years, the problem of pollution has highlighted the need for urgent research. Environmental literature on Pakistani distillery wastewater is scarce; therefore, research on foreign distillery industries should consider the proposed solutions. Some mills pretreat wastewater, but do not meet the required discharge standards. Other sectors use additional methods, such as aerated lagoons, to improve effluent quality for agricultural use. However, countries such as India, Thailand, Brazil, and South Africa have similar situations regarding distillery wastewater disposal. The governments of these nations have implemented strategic policies and have executed comprehensive industrial solutions to handle comparable challenges. Table 5 summarizes the comparative analyses.

5. Future research directions in nanomaterials for wastewater treatment in Sindh, Pakistan

A review of critical research gaps and opportunities for nanomaterial-based wastewater treatment in Pakistan to address resource-constrained and environmental degradation problems. Four key research priorities have emerged:

Table 5. Comparative analysis between Sindh and global practices for distillery wastewater remediation.

Aspect	Sindh (Pakistan)	Global practices
Primary Treatment	- TSS is removed by sedimentation 30–50% (Khan <i>et al.</i> , 2003). - There is no autotossation and neutralization (lime/alum) adjust the pH.	- Automated systems have TSS (70–90%) removal (EPA, 2020). - Dosing with real time suggests a consistent pH control.
Anaerobic Treatment	- 40–60% removal of COD: 1–2 hectares required per 1,000 m ³ (Rasool <i>et al.</i> , 2023). - Biogas yield in the region of 0.1–0.2 m ³ /m ³ wastewater	- 80–90% removal of COD: less than 0.1 hectares required per 1000 m ³ (Fito <i>et al.</i> , 2018). - Biogas yield of 0.3–0.5 m ³ /m ³ (with energy recovery).
Biological Treatment	- 60–75% of COD removal: wastewater native microbial communities. (Ali <i>et al.</i> , 2024).	- 85–95% of COD removal: Engineered strains + hybrid systems (Ravikumar <i>et al.</i> , 2021). - 71.83% color removal (Chowdhary <i>et al.</i> , 2020)
Constructed Wetlands	- 70% of COD removal: Pilot studies (Rehman <i>et al.</i> , 2024). - Lack of Policy support and underutilization	- 85–95% of COD removal: Large-scale systems (Zurita & Vymazal, 2023). - Cost-effective and localized method for distributed treatment.
Physico-Chemical	- 85% of COD & 91% color removal: Electrocoagulation (Khokar <i>et al.</i> , 2023). - Expansive operational method (\$0.5–1/m ³).	- > 90% of COD and color removal: Advanced oxidation (Khandegar & Saroh, 2014). - Economical operational method (\$0.3–0.8/m ³)
Membrane Tech.	- Small scale biogas plants: < 50% COD with >\$200,000 capital expenses (Parveen & Khan, 2023).	- Industrial scale MBR/RO systems: 90–95% COD removal with 80% water recovery (Gupta <i>et al.</i> , 2008).
Energy Recovery	- less than 10% energy recovery: Biogas rarely encapsulated (Jatoi <i>et al.</i> , 2018).	- More than 50% energy recovery: CHP systems with 0.45 to 0.55 m ³ /Kg of Biogas production. (Wagh & Nemade, 2018).
Sludge management	- 30–50% dumped without treatment (Kaloi <i>et al.</i> , 2017).	- 90% waste is treated using pyrolysis and gasification methods (Dhote <i>et al.</i> , 2022).
Water reuse	- Less than 20% reused: majority being discharged as untreated (Noor <i>et al.</i> , 2023).	- 50–70% reused: mostly for irrigation or industrial use (Kharraz <i>et al.</i> , 2022).
Pollutant Removal	- COD effluent range: 300 to 500 mg/L (250 mg/L set by the U.S. Environmental Protection Agency). (Nergis <i>et al.</i> , 2012)	- COD effluent range: <50 mg/L (Using Integrated MBR+RO systems) (Dialynas Diamadopoulos, 2009)

5.1. Development of multifunctional nanocomposites and membranes

Engineered nanocomposites (e.g., TiO₂/graphene oxide hybrids or MOFs/polymer hybrids) in which adsorption and degradation by catalysis and membrane filtration effect are synergized remain to be investigated in future work. By utilizing such materials, dyes, heavy metals, etc. can be removed effectively (>90%) with regenerability and reduced fouling. To enable industrial adoption, it must be scalable and analyzable by lifecycle.

5.2. Utilization of agricultural waste for sustainable nanomaterials

The sustainable feedstock used for low-cost nanocatalysts and biochar includes agricultural residues (e.g., rice husk silica and date palm-activated carbon). The protocol for pyrolysis/functionalization should be optimized based on research to enhance the pollutant affinity (e.g., Cr (VI)) and ecotoxicity of the byproducts. Recycling garbage sources aligns with SDG 12 (Responsible Consumption).

5.3. Solar-activated photocatalysis for decentralized systems

In Sindh, where the solar irradiation is approximately 2200 kWh/m²/yr, photocatalytic nanomaterials (such as ZnO/SnS₂ heterojunctions) can generate water in an off-grid treatment regime with a sustainable load. Quantum efficiency under natural sunlight remains a critical challenge, and photocorrosion issues need to be resolved. Long-term performance in rural settings with high organic loads must be evaluated in pilot studies.

5.4. Integration of smart nano-sensors for real-time monitoring

Coupling Au/Ag or fluorescent nanoparticles into IOT-enabled sensors for monitoring COD, heavy metals, and pathogens could revolutionize integrated monitoring. Underfunded municipal facilities require low-cost production (e.g., paper-based tests) and choose research in difficult matrices to use these technologies.

Future research will need to resolve the databases of realistic risk assessments of the mechanisms of interaction of real-world nano-pollutants with environmental pollutants. Furthermore, international collaboration and interdisciplinary work in environmental science, chemical engineering, and materials chemistry are necessary for the development of scalable and cost-effective treatment technologies. This will enable Universities, Industries, and Governmental agencies to accelerate the deployment of these sustainable solutions in Pakistan through Pilot Programs and Funding Initiatives.

6. Potential socio-economic benefits of adopting nanomaterial-based treatment methods in Sindh, Pakistan

The integration of advanced materials in wastewater treatment offers significant cost advantages by effectively reducing contaminants to negligible levels in the initial applications. Because of their improved activity, selectivity, and reusability, the use of small amounts of these materials results in significant cost savings and low spatial requirements, making their wider adoption possible. These technologies provide affordable treatment options; ensure public safety; and provide jobs in the manufacturing, installation, and system operation industries. Enhanced wastewater treatment improves public health by reducing the occurrence of waterborne infections and improving the water quality. The application of advanced materials such as nanomaterials for efficient contaminant removal or transformation extends the socioeconomic benefits of water reuse and agricultural productivity. Over 6,000 full-time jobs can be created by treating and disposing of 10% of used agricultural water, demonstrating the substantial employment contribution of the water sector. Productivity gains, regardless of how little, can boost job prospects, particularly for low-income groups. Because cleaner water resources support jobs in a range of industries, including agriculture, fishing, and water and sanitation services, their economic value has increased. For every 1% increase in population access to improved water supply, agricultural productivity might increase by 0.7%. Social and economic structures are affected by geopolitical and economic interconnections driven by modern global dynamics.

7. Conclusions

Enhancing distillery wastewater treatment procedures using nanoparticles has become a viable way to address the intricate problems of resource management and water contamination. The primary benefits of nanomaterials are highlighted in this review, including their ability to increase the pollutant removal efficiency through adsorption. In addition, nanomaterials contribute to resource recovery and environmental sustainability, offering opportunities for their incorporation into broader water-management strategies. However, successful adoption of these technologies requires a balanced approach that considers environmental, economic, and regulatory factors. Increasing scientific understanding, encouraging teamwork, and supporting creative research will be essential for achieving the full potential of nanomaterials in distillery wastewater treatment and for assisting sustainable water management techniques for future generations.

Author Contributions

Mehwish Qaseem: Contributed to structuring the manuscript outline, conceptualizing the research theme, conducting an extensive literature review, and preparing the original draft.
 Yasir Khan: Provided constant guidance during the study and reviewed the final manuscript.
 M. Waseem Akhter: Worked with the comparative analysis of local versus global treatment methodologies and evaluated their performance.

Syed Zeeshan Abbas: Ensured journal-compliant formatting and offered insights into the importance of the topic in environmental policies.

Mehwish Altaf: Performed a literature review on the features of distillery wastewater and collected pertinent data from Sindh, Pakistan.
 Shakeel Ahmed: Focusing on the nanomaterial adsorbents section, their properties, and mechanisms of adsorption in wastewater treatment.

Muhammad Saquib Ali: Supported the manuscript with linguistic consistency and citation management.

Conflict of Interest

There are no conflicts to disclose.

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Data Availability Statement

The data for this study are available within the article and can be obtained from the corresponding author upon request.

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