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Nickel Remediation by Adsorption Technique Achieving the Concept of Zero Residue Level

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ABSTRACT

Nickel is a major threat to the wastewater discharged from industries, where it is used, because it is classified as a carcinogenic heavy metal. Due to the need to treat polluted water with the highest efficiency and lowest cost, the promising adsorption technology using agricultural waste has proven to be an effective solution for treating water pollution with various pollutants. This study is concerned with determining the ability of a household waste to remove nickel from contaminated aqueous solutions by the adsorption method at different operating conditions. Waste tea leaves (WTL), which are discarded in huge quantities annually, showed noticeable nickel removal efficiency at more than 74% at acidity, shaking, concentration, time, temperature, and adsorption dose of 6, 350 rpm, 42 ppm, 120 min, 20°C, and 5.5 g, respectively. Although the adsorbent used had a low surface area of 12.251 m^2/g, FTIR test before and after adsorption confirmed the presence of various functional groups capable of efficiently performing the adsorption function. Furthermore, FESEM test showed that the adsorption medium suffered significant changes compared to what it was before treatment with contaminated solutions. The use of toxic adsorption residues in a beneficial way was tested by adding them to concrete mixtures and studying the compressibility performance. The results showed that the additives increased the compressive strength of the concrete by more than double before it reached the failure point. Thus, this investigation confirmed the possibility of achieving the concept of zero residue level through waste management in an eco-friendly manner.

Keywords: Adsorption, Concrete additives, Nickel, Waste tea leaves, Zero residue level.

INTRODUCTION

Environmental pollution is any undesirable change in the physical, chemical, or biological properties of the environment surrounding humans and other living organisms, resulting from human activities or natural processes, leading to negative effects on the health of living organisms, the balance of ecosystems, and the quality of natural resources, such as air, water, and soil (Khaleel et al., 2022). Pollution can be visible and obvious, such as water pollution by waste, or invisible, such as radioactive pollution or air pollution by gases (Abbas & Ibrahim, 2020). Pollution involves the introduction of pollutants, which are substances or energy negatively affecting the environment (Al-Zboon, 2023). Environmental pollutants include a wide range of substances and activities that negatively affect the environment and the health of living organisms (Abbas & Abbas, 2013a). The most important types of these pollutants are heavy metals. Heavy metals are defined as elements that have a density of at least five times higher than that of water and include quite a few elements (Abbas & Abbas, 2013b). Although they are indispensable in industrial, agricultural and other human activities, and some of them are even needed by humans, they are considered among the most dangerous types of pollutants due to their toxic and cumulative effects on the environment, and some of them are classified as carcinogenic to living organisms (Ali et al., 2021). Heavy elements are used in the manufacture of batteries, paints, dyes, pipes, mining, pesticides, alloys, medicines, medical preparations, electronics, catalysts, … etc. (Ali & Abbas, 2020). At the same time, these elements lead to many negative effects on the environment and human health if their concentrations exceed the permissible limits (Alhamd et al., 2024a). Many studies have proven that exposure to heavy metals at different times and concentrations can cause many health problems, including liver and kidney failure, osteoporosis, skin allergies, respiratory diseases, and cardiovascular diseases, in addition to being one of the main causes of various types of cancer (Kim et al., 2015). One of the most important and widely used heavy metals is nickel. It is a chemical element that has many important uses in various industries due to its unique physical and chemical properties (Maddodi et al., 2020). One of the oldest uses of this metal is in the manufacture of coins, and it is used in the synthesis of alloys to improve their resistance to corrosion, rust and heat, such as stainless steel (Alsarayreh et al., 2024). It is also widely used in electroplating processes to give products an attractive appearance and to protect them from corrosion (Genchi et al., 2020). It is also an essential component in the manufacture of batteries, such as nickel-cadmium (NiCd) and nickel-metal hydride (NiMH) batteries, which are widely used in portable devices, cell-phone batteries and digital cameras (Shen et al., 2020). In addition to the above, nickel is an essential component as a catalyst in many chemical processes, such as petroleum refining and the production of organic chemicals (Genchi et al., 2020). In the medical field, nickel is used in the manufacture of surgical instruments and some medical devices due to its high resistance to chemical corrosion and biological corrosion (Chou et al., 2013). Despite its great industrial benefits, nickel is considered toxic and has negative health effects if humans are exposed to it in large quantities. Inhaling dust or fumes containing nickel can lead to severe respiratory problems, such as rhinitis, occupational asthma, and pulmonary fibrosis (Genchi et al., 2020). Long-term exposure to nickel affects kidney function and increases the risk of cardiovascular disease. More seriously, it can increase the risk of lung, throat, and sinus cancers (Lee et al., 2021). Due to the negative effects of this element, the maximum permissible concentration of nickel in drinking water is 0.02 mg/L according to the World Health Organization (WHO), and the US Environmental Protection Agency (EPA) (Wołowicz et al., 2021), which is the same percentage listed in the Iraqi specification (IQS-417 2001). Exceeding this percentage can be dangerous to public health, so removing this metal from water or reducing its concentration to the minimum possible level is an urgent necessity to preserve human and environmental safety at the same time (Lee et al., 2021). There are many methods used to treat water contaminated with heavy metals, including ultrafiltration, ion exchange, chemical precipitation, coagulation-flocculation, advanced oxidation process, chemical reduction, electrolysis method, biological treatment, and adsorption (Abbas et al., 2020). These methods are highly efficient in removing heavy metals from polluted water, but they also have a number of drawbacks depending on the nature of the polluted water, the type and concentration of heavy metals, the costs associated with the process, the consumption of energy, pre-treatment, special equipment and systems, the space required to carry out the process, and the time of the process (Ali et al., 2024a). Among the above methods, the adsorption method stands out as one of the efficient and economical methods for treating polluted water, as this technology does not require large costs or high energy consumption, in addition to the fact that it does not require a large area, special equipment, or pre-treatment (Alhamd et al., 2024b). Moreover, this technology is highly efficient in treating many types of wastes, such as dyes (Alalwan et al., 2021), organic acids (Abbas & Abbas, 2014), inorganic toxics (Alalwan et al., 2020), water hardness (Ibrahim et al., 2021), radioactive elements (Alalwan et al., 2018), eutrophication elements (Abbas, 2015), heavy metals (Shadhan et al 2024), pesticides (Abd Ali et al., 2024), pharmaceuticals (Ibrahim et al., 2020a), drugs (Ibrahim et al., 2020b), especially if materials such as activated carbon (Alhamd et al., 2024c), zeolite (Khudair et al., 2024), biochar (Qasim et al., 2023) and alumina are used as effective adsorption media (Ghulam et al., 2020). Studies have shown that adsorption technology is highly efficient in removing pollutants, not only from water, but also from soil (Abbas et al., 2021), and crude oil (Abbas & Alalwan, 2019). Although the adsorption technology has the characteristics of efficiency and economy, the high cost of the adsorption media and the need for continuous regeneration, in addition to losing some weight in each regeneration process, are major obstacles that prompted researchers to search for alternatives to these materials (Safaei et al., 2023). Here, low-cost, negligible toxicity of agricultural and industrial waste emerged as one of the solutions facing this promising technology, as this waste represents a heavy burden on the environment due to its disposal in huge quantities and the accompanying ongoing environmental problems (Abbas et al., 2019a). Among these wastes that have proven to be highly efficient in their use as adsorption media are pomegranate peels (Ali et al., 2024b) rice husks (Abbas & Abbas, 2013c), banana peels (Abdullah et al., 2023), watermelon rinds (Abbas and Nussrat, 2020), eggshells (Ali et al., 2020a), waste tea leaves (Al-Ali et al., 2023), lemon peels (Al-Hermizy et al., 2022), orange peels (Hasan et al., 2021), tree leaves (Hameed & Abbas, 2024), water hyacinth (Hashem et al., 2021), algae (Abbas et al., 2019b), aluminum foil (Ghulam et al., 2020), among others. However, their accumulation after the end of the treatment process causes a real problem due to their high toxicity. This problem can be solved by applying the concept of zero residue level, which considers these wastes as materials that can be used in an economical way instead of disposing of them using expensive methods (Alhamd et al., 2024d). Through a sustainable zero-residue level approach, these wastes have been converted into useful chemicals, such as acetone (Abbas et al., 2022b), bioethanol (Hamdi et al., 2024), soil fertilizer (Abbas, 2015), rodenticides for rats (Abd Ali et al., 2018) and rabbits (Abd Al-Latif et al., 2023), catalysts (Abbas et al., 2021), nanomaterials (Alminshid et al., 2021), and concrete reinforcement additives. Previous studies have shown that the use of lead-laden rice-husk waste increased the compressive strength of concrete while reducing the lead layers used in the construction of hospital X-ray rooms (Abbas et al., 2022a). On the other hand, a study of Abdulkareem et al. (2023) proved that adding sunflower husks in specific proportions to concrete reduced its weight and enhanced its compressibility, a result similar to that of the study of Rajaa et al. (2023), who used copper-loaded tea leaves as an additive to the concrete mixture. The present study aims to apply a sustainable zero-residue level approach by using waste tea leaves in two stages: the first as a low-cost adsorbent to remove nickel from contaminated aqueous solutions in a batch-type unit under different operating conditions, and the second using the nickel-loaded remnants as an additive to concrete mixtures to enhance compressive strength.

METHODOLOGY

Adsorbent Preparation: The adsorbent employed in this study is waste tea leaves obtained from the remains of daily domestic consumption of tea beverage. The cleaning process of the leaves involved multiple washes with tap water followed by rinsing with deionized water, ensuring that any dirt or adhering impurities were removed to obtain a clean and pure material. The leaves then underwent a drying process, first in sunlight for several days, followed by a period in a hot-air oven at 40°C for 10 hours. A 100-g sample of dried used tea leaves was sieved through sieves ranging from 35 to 60 mesh. The final dried powder was stored in opaque amber-glass bottles. The sieve-analysis results of the waste tea leave powder used in this study are presented in Table 1.

Table 1. Sieve analysis of waste tea leaves powder used in this study

#	Mesh	Sieve Size, (μm)	Weight (g)	% wt
1	35	500	17.58	17.58
2	40	400	9.43	9.43
3	45	354	19.44	19.44
4	50	297	47.92	47.92
5	60	250	3.02	3.02
7	Pan	-	2.61	2.61
Σ	-	-	100 g	100%

Zero Charge Point (pH_{pzc})

The pH_{PZC}, an important parameter controling the reactivity and adsorption behavior of the adsorbent, was determined using the solid-addition method—a fundamental technique in surface chemistry and adsorption studies. This method ascertains the pH at which the surface of the adsorbent carries no net charge. A stock solution of 0.01 N potassium nitrate was prepared using 1.013g of KNO_3 (99.8%, sourced from MERCK-Sigma-Aldrich-Germany) by a digital analytical lab scale (Precision Balance – USS-DBS81-110G-US Solid). The determined mass of potassium nitrate was then dissolved in 1000 cm^3 of double-distilled water. The dissolution process was carried out under laboratory conditions with gentle manual mixing until dissolving all KNO_3 mass. Thus, the resulting 0.01 N KNO_3 solution was stored in amber-glass bottles of suitable capacity using glass funnel. The pH_{PZC} measurement was determined using the solid-addition method. This method ascertains the pH at which the surface of adsorbent carries no net charge. A series of 100-cm^3 flasks were employed to prepare suspension solutions. In each flask, 100 cm^3 of 0.01 N KNO_3 solution was put, and the pH of each solution was measured using (HI-83141-1 pH Meter, Hanna-USA). The pH was adjusted to the desired value by 0.1 N HCl or NaOH solutions. The pH of flasks ranged 1-14 and these numbers were considered as the initial pH values. Subsequently, one gram of waste tea leaves samples (50 mesh) was added to each flask under 100-rpm stirring at ambient temperature overnight using bath shaker (Marshall Scientific Barnstead MaxQ 7000). Following a 24-hour equilibration period, the average pH values were determined based on five separate measurements of the liquid supernatant. These measurements were conducted to ensure the accuracy and reliability of the obtained pH_{PZC} value.

Preparation of Nickel Stock Solution: The stock solution of nickel metal was formulated using a package of Extra Pure - 500g nickel nitrate hexahydrate which has a formula $Ni(NO_3)_2.6H_2O$, obtained from HiMedia company, India. For solution preparation, an amount of 5 g of emerald green powder nickel salt was dissolved in 100 cm^3 of double-distilled water, prepared *via* a water distillation unit (GFL-2012). Employing a magnetic stirrer under laboratory conditions, the mixing process persisted until a homogeneous solution was achieved. This precise method ensured that each cubic centimeter of the solution contained 1 mg of nickel metal, resulting in a solution of 1,000-ppm concentration.

Calibration Curve of Nickel: To find the nickel concentration in the aqueous solutions, before or after adsorption, Atomic Absorption Spectroscopy (AAS) instrument (Shimadzu AA-7000, Japan) was employed at a wavelength of 341.5 nm. A calibration curve for nickel ions was prepared according to the procedure described by Ali et al. (2020b) *via* analyzing several samples of known concentrations of nickel, correlating each concentration with its corresponding absorbance value. Figure (1) shows the calibration curve of nickel achieved *via* AAS analysis used in this study.

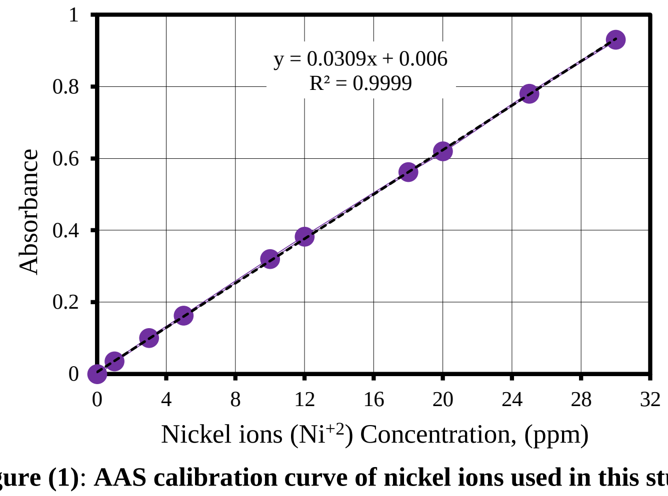

Figure (1): **AAS calibration curve of nickel ions used in this study**

Adsorption Unit Used: The adsorption experiments of nickel metal using waste tea leaves adsorbent were conducted in a batch mode unit represented by a Water Bath Shaker of type (Thermo Scientific MaxQ 7000 SHKE7000 Benchtop Model 4303, 2011, USA). A desired concentration of nickel metal was prepared by diluting the stock solution to the required one. Various operating parameters including pH, agitation speed, initial concentration, waste tea leaves dose, contact time, and temperature were studied. The pH of the solution was controlled using 0.1 N sodium hydroxide (NaOH) and 0.1 N hydrochloric acid (HCl) solutions. The ranges of parameters tested were varied among (1-9), (100-500 rpm), (1-50 ppm), (10-180 minutes), (20-50ºC), and (0.5-6.0 g) for pH, agitation speed, initial concentration, waste tea leaves dose, contact time, and temperature, respectively. The experiments were designed by adding 100 cm^3 of the nickel solution of desired concentrations and pH values, to a sealed conical flask of appropriate capacity. The flask was then placed in a water bath shaker, and the experiment was commenced by setting the required agitation speed. The experiment duration was determined by the pre-defined contact time. After completion, the solution was filtered using a vacuum filtration apparatus (JOANLAB VP-10L), after separating the waste tea leaves from the slurry. The remaining concentration of nickel metal in the treated solution was measured using an AAS device. The nickel percentage removal was calculated via Equation 1, while the adsorption capacity of the waste tea leaves was obtained from Equation 2.

$$\boxed{\%R = \left(1 - \frac{C_f}{C_\circ}\right) \times 100} \qquad \ldots (1)$$

$$\boxed{q = \frac{V}{1000}\frac{(C_\circ - C_f)}{m}} \qquad \ldots (2)$$

where: $\%R$ is the removal efficiency of nickel metal; $C_\circ$ is the initial concentration (ppm); C_f is the final concentration (ppm); q is the adsorption capacity of waste tea leaves for nickel metal, expressed in (mg/g); V is the volume of the solution used (ml); and m is the mass of waste tea leaves (g).

RESULTS AND DISCUSSION

Characterization of Adsorbent Media

BET (Brunauer, Emmett, Teller) surface area (S_{BET}): The analysis of the surface-area values of waste tea leaves was conducted using Quantachrome, Qsurf 9600 Thermo Finnegan Co., USA device. The values determined were 12.251 m²/g and 1.152 m²/g before and after nickel-metal adsorption, respectively, as in the adsorption-desorption plot shown the Figure (2). The notable reduction in surface area of waste tea leaves from 12.251 m²/g before nickel-metal adsorption to 1.152 m²/g after adsorption offers valuable information about the adsorption mechanism and relates well with the observations made during the FTIR and SEM analyses. The initial high surface area of waste tea leaves suggests the presence of a porous structure. The surface area of waste tea leaves after adsorption of nickel metal was reduced to about 9.4% of the original value. This intense decline indicates that a great portion of the surface area of the leaves has been occupied by nickel ions. These interactions likely played a pivotal role in the binding of nickel ions to the adsorbent, which absolutely led to the roughness structure after adsorption and reduced the surface area.

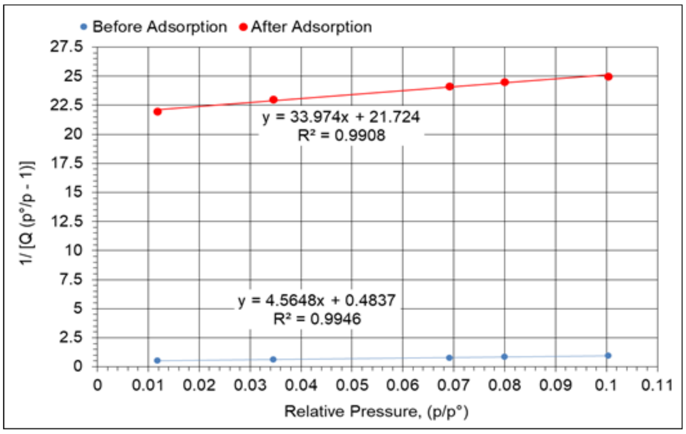

Figure (2): **Adsorption-desorption isotherm of waste tea leaves**

Fourier Transform Infrared Spectroscopy (FTIR) Test: Using FTIR spectrophotometer (IRPrestige-2, Shimadzu, Japan), the FTIR spectra of waste tea leaves before and after adsorption of nickel metal are presented in Figure (3). These spectra offer a valuable view into the structural changes and chemical interactions within the material. Prior to adsorption, the FTIR spectrum displayed distinct peaks at various wavenumbers, providing evidence about the composition of the waste tea leaves. After nickel adsorption, notable changes in the FTIR spectrum were observed. Shifts and new peaks are appearing, indicating the interaction between nickel and waste tea-leaves. The changes in the FTIR spectrum of tea leaf residues before and after adsorption of nickel ions clearly indicate that chemical reactions took place between the components of tea leaves and nickel ions. These reactions involve the formation of new bonds or the modification of existing bonds, resulting in changes in the characteristic stretches of different functional groups in the FTIR spectrum.

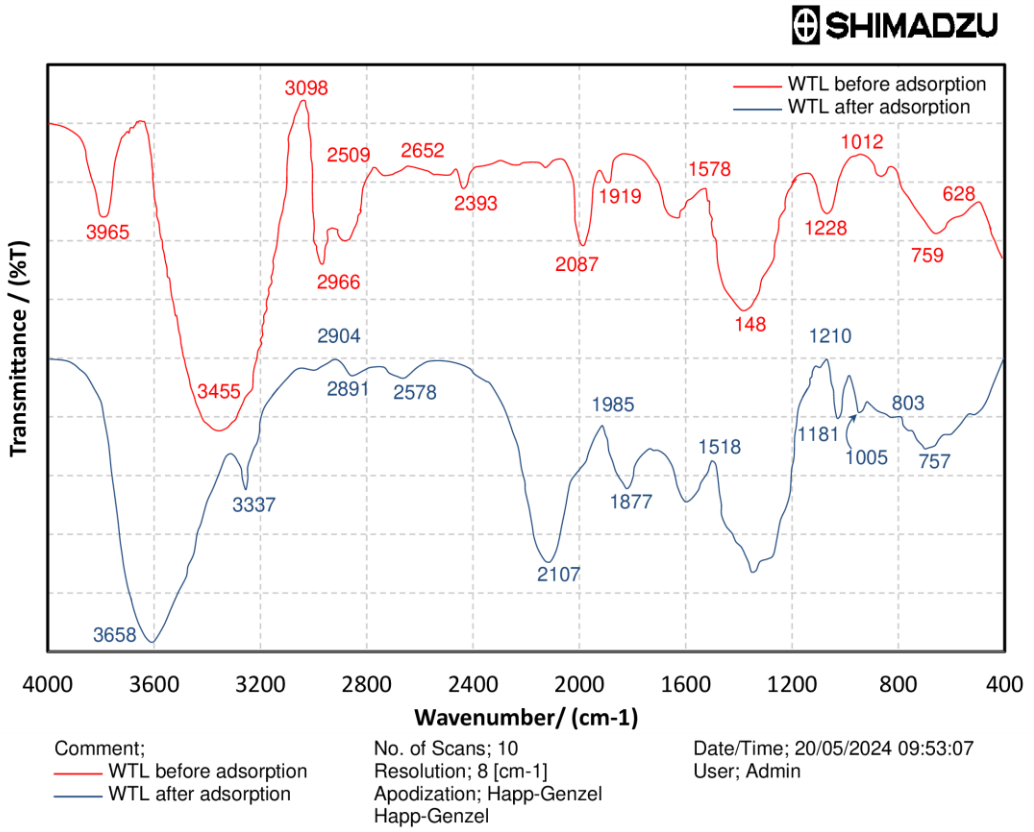

Figure (3): **FTIR spectrum of waste tea leaves**

Field Emission Scanning Electron Microscopy (FESEM) Test: The surface morphology of the adsorbent can be demonstrated by field emission scanning electron microscopy (FESEM) images. In the present study, the micrograph of waste tea leaves before nickel adsorption showed a smooth and homogeneous nature. However, after adsorption of nickel metal, the surface is almost heterogeneous, rough and porous (Figures 4a, b). The transformation of adsorbent surfaces can be attributed to the process of adsorbing nickel ions. As the metal ions interact with the waste tea leaves surface, they adhere to its irregularities and porous structures. This interaction leads to a coating effect, where the metal ions fill in the gaps and unevenness present on the surface. Consequently, the previously smoother and more uniform appearance becomes coated with a layer of dye, resulting in a non-uniform surface. This phenomenon is a manifestation of the molecular-level interactions between the nickel ions and the waste tea leaves surface, contributing to altering its physical characteristics. However, after nickel-metal adsorption, the surface of waste tea leaves peel is rough and porous in nature.

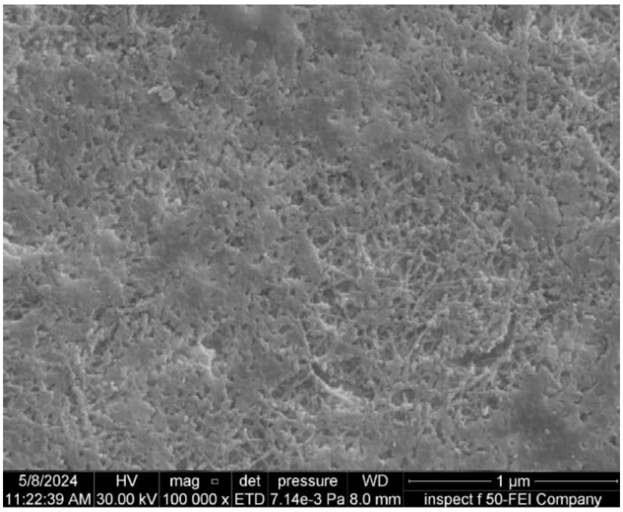

Figure (4a): FESEM of waste tea leaves before adsorption of nickel

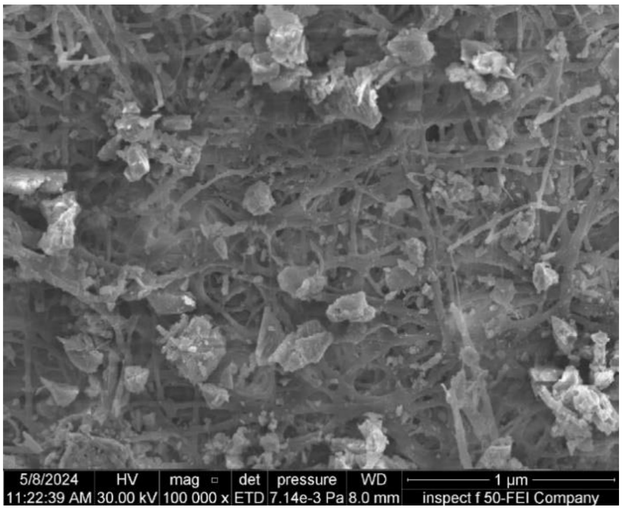

Figure (4b):FESEM of waste tea leaves after adsorption of nickel

Point of Zero Charge (pH_{PZC}): Alkali metal nitrate, especially potassium nitrate (0.01 N), is usually used to estimate the point of zero charge. The point of zero charge of waste tea leaves pH_{ZPC} was determined, and the results obtained are shown in Figure (5). The pH_{pzc} values were known for determining the position where the resulting curves cut through the pH_o axis. For waste tea leaves, pH_{pzc} was recorded as 6.979. This value can be useful for predicting the surface charge of the adsorbents; when adsorption occurred at a particular pH which is below the point of zero charge of the adsorbent, the surface of the adsorbent will be positive, and when adsorption occurred above the point of zero charge pH, the adsorbent surface charge will be negative. The pH *versus* pH_{PZC} of the waste tea leaves is depicted in Figure (5).

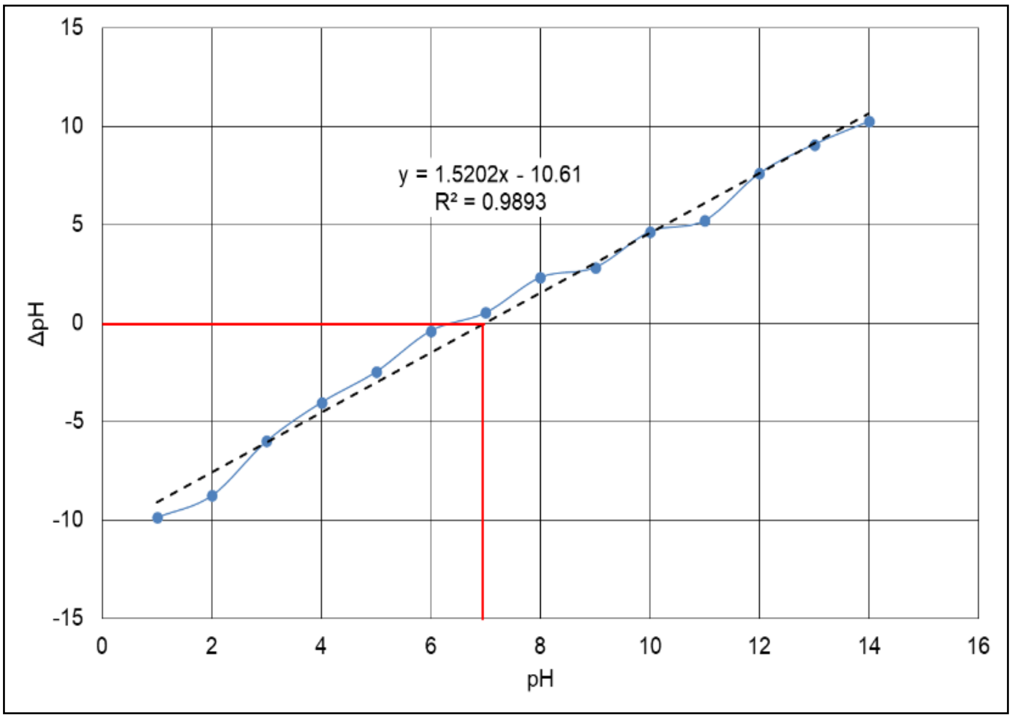

Figure (5): pH_{PZC} of the waste tea leaves

Acidity Impact: Figure (6) explains the effect of changing the acidity, in the range from 1 to 9 on the percentage removal of nickel ions from polluted aqueous solutions using waste tea leaves as a low-cost adsorbent, while maintaining other operational conditions at constant values of 1 ppm initial concentration, 180 minutes of contact time, 1 g of adsorbent dose, 300 rpm of agitation speed, and 20°C of temperature. As shown in Figure (3), the remediation efficiency displays a gradual increasing with rising solution acidity, starting at 7.33% at pH 1 and peaking at 38.175% at pH 6. This result can be explained by the positively charged surface of the adsorbent, which is caused by hydrogen ions at low pH values. This positive charge generates a strong repulsive force between the surface of waste tea leaves and the ions of nickel metal in the solution, which leads to a decrease in the removal efficiency. Further, the excess hydrogen ions in the solution compete with nickel ions for the available active sites on the surface of waste tea leaves. Conversely, high pH values reduce the positive charge on the adsorbent surface, which reduces the resistance to adsorption of nickel ions, and thus increases the efficiency. When the pH value was increased beyond 7, two significant notes were observed. First, the removal efficiency increased sharply and rapidly until it reached 100%. Second, the nickel ions precipitated at the bottom of the experimental flask. This phenomenon indicates that the increase in removal efficiency to more than 38.175% is due to the precipitation process rather than to adsorption. In the precipitation process, nickel ions remain in the solution, even with restricted diffusion, and the removal efficiency reaches its peak. However, this behavior does not equate to the removal of the target ions, but only neutralizes their activity. Thus, the optimum acidity value of nickel adsorption using waste tea leaves is 6 (Ibrahim et al., 2021).

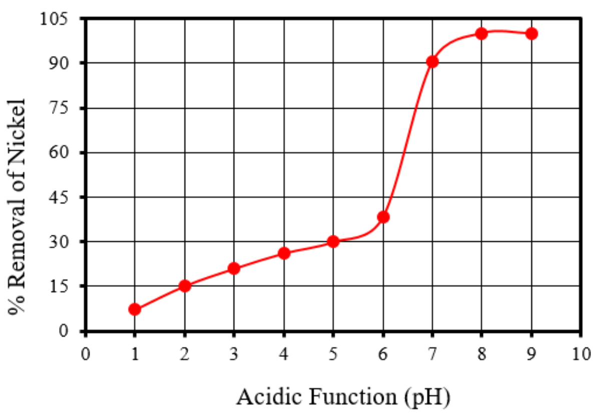

Figure (6): **Acidity impact on the nickel removal**

Agitation Speed Impact: Figure (7) exhibits the effect of agitation speed on the percentage removal of nickel metal using waste tea leaves as a low-cost adsorbent, while keeping other parameters constant at 9 of pH, 1 ppm of initial concentration, 1 g of waste tea leaves, 180 minutes of contact time, and 20°C of temperature. According to the results shown in Figure (7), the percentage removal of nickel ions increased from 10.153% to 40.402% as the agitation speed increased from 100 rpm to 350 rpm, respectively. This result can be attributed to many explanations. Firstly, the raising of shaking may have enhanced the mass transfer between the waste tea leaves and nickel ions in the solution, leading to increase the rate of adsorption. This may lead to more efficient contact between the waste tea leaves and nickel ions in the solution, resulting in higher percentage removal. Secondly, the increasing of shaking assists to fragmentize any film layer that formed around the waste tea leaves, ensuring fresh solution coming into contact with the adsorbent surface. This prevents the formation of stagnant regions and promotes better adsorption. Further, the high shaking can dislodge adsorbate molecules initially adsorbed onto the surface of waste tea leaves, making more adsorption sites available. However, beyond 350 rpm, the percentage removal remained constant. This can be explained as the adsorption sites on the adsorbent surface becoming filled and saturated over time, reaching their limit. Once the adsorption sites are filled, the percentage removal remains constant regardless of the agitation speed. Thus, the optimum agitation speed value of nickel adsorption using waste tea leaves is 350 rpm (Ali et al., 2023).

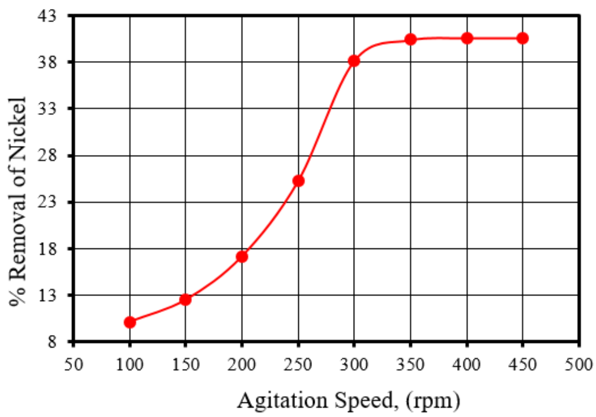

Figure (7): **Shaking impact on the nickel removal**

Initial Concentration Impact: To check the effect of nickel initial concentration, a set of experiments were performed using 1 g of waste tea leaves. The acidity, agitation speed, contact time, and temperature were kept constant at 6, 450 rpm, 180 minutes, and 20°C, respectively. The range of initial concentrations studied was changed from 1 ppm to 50 ppm. The results showed an inverse relationship between the percentage removal and the initial concentration of nickel metal. The maximum percentage removal of 40.402% was achieved when the initial concentration was 1 ppm, while at 50 ppm, the percentage removal decreased to 16.784%. This inverse relation can be attributed to the concept of adsorption equilibrium. At higher initial concentrations, there are more ions of nickel available for adsorption onto the surface of waste tea leaves, leading to a faster saturation of the active sites. As a result, the removal efficiency decreases as the initial concentration increases. Figure (8) shows that the adsorbed concentration increased until reaching 42 ppm and then remained constant. This indicates that the waste tea leaves became saturated, as all available sites on the surface of waste tea leaves were occupied by nickel ions, forbidding any additional adsorption (Alwan et al., 2021).

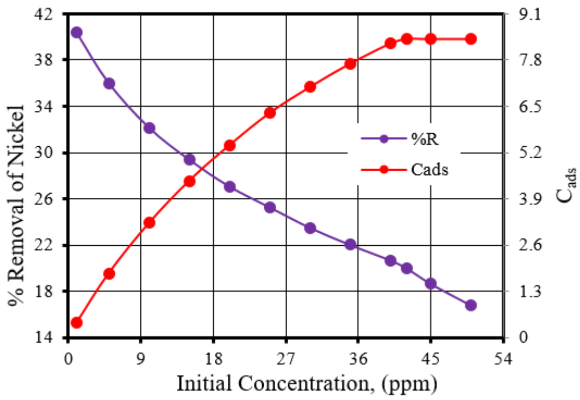

Figure (8): **Initial concentration impact on the nickel removal**

Adsorbent Dosage Impact: The impact of adsorbent quantity on the adsorption of nickel was explored under values of optimum parameters which were 6, 350 rpm, 42 ppm, 180 minutes, and 20°C, of acidity, shaking, initial concentration, contact time, and temperature, respectively. The results, as displaced in Figure (9), disclosed that the percentage removal of nickel increased by increased dosage of waste tea leaves as a low-cost adsorbent. This result suggests that increasing the adsorbent dosage enhances the ability for recovering the nickel ions from the contaminated solution. This can be explained by the increased availability of active sites on the surface of waste tea leaves, carried out as the mass is increased. With more active sites available for adsorption, there is a greater chance for nickel ions to bind to the adsorbent, resulting in amending the percentage removal (Abdulhammed & Alhamd, 2024).

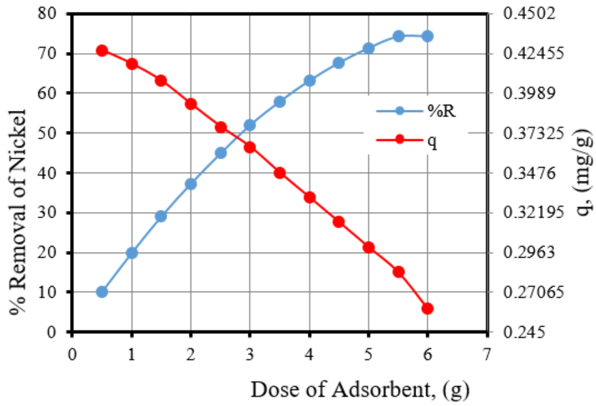

Figure (9): **Adsorbent dose impact on the nickel removal**

Contact Time Impact: The effect of contact time on the removal efficiency of nickel ions was studied by keeping other factors fixed at constant values. The results illustrate that rising the contact time from 10 minutes to 180 minutes leads to a considerable increase in the removal efficiency of nickel ions from 16.516% to 74.301%, as elucidated in Figure (10). This essential amendment in percentage removal may be due to that the longer time allows more contact between the nickel ions and the waste tea leaves. As the contact time increases, there is more opportunity for the occurrence of the recovering of pollutant by the adsorption process, resulting in a high efficiency of nickel being removed from the solution (Adnan et al., 2023).

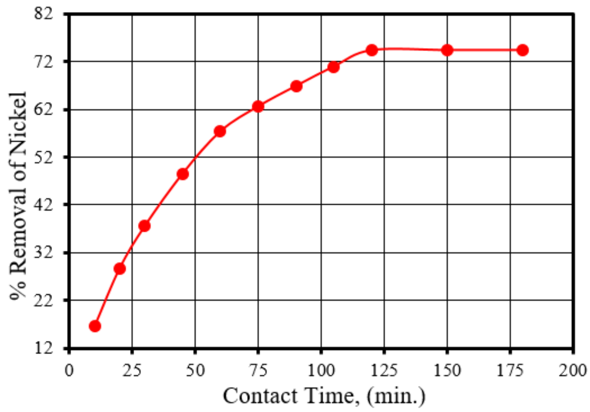

Figure (10): **Time impact on the nickel removal**

Temperature Impact: The effect of temperature on the percentage removal of nickel was recognized in this study, revealing a notable decrease in removal efficiency from 74.301% to 10.153% as the temperature increased from 20°C to 50°C, as shown in Figure (11). Higher temperatures can disrupt the physical adsorption process by causing weaker interactions between the adsorbent material and the nickel ions. When temperature rises, the increased thermal energy can weaken the attractive forces between the active sites of adsorbent media and the nickel ions, resulting in a reduced adsorption capacity and hence in a lower efficiency of nickel removal (Alhamd et al., 2024a).

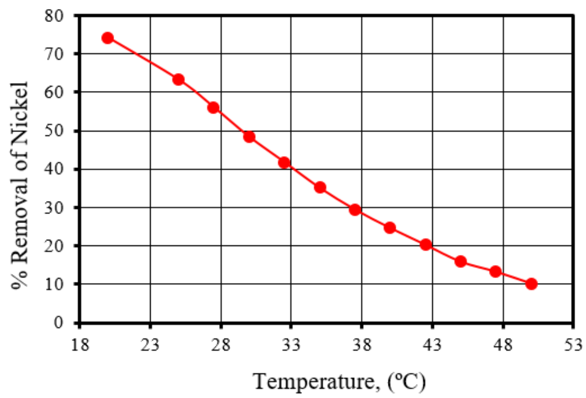

Figure (11): **Temperature impact on the nickel removal**

Utilizing the Ni^{+2}-waste Tea Leaves: After determining the efficacy of waste tea leaves as a low-cost adsorbent for nickel ions in aqueous solutions, the later step involved the safe disposal of this toxic-loaded remnants using an eco-friendly method. One suggested method was the utilization of these toxic residues as an additive to concrete mixtures to enhance the performance of concrete mixtures *via* measuring the compressive strength before and after addition (Khan & Sharma, 2018). To achieve this procedure, three types of concrete mixtures were prepared, the first one is a reference mixture with mixing ratios of (4:2:1) (gravel:sand:cement) and a ratio of 0.45 (water to cement), and the other type is a mixture with the same previous proportions, with adding 1.8% of virgin waste tea leaves by weight. The last type was the same second type, but with adding Ni^{+2}-waste tea leaves by ratios that ranged between (0.1-2.8) % of the mixture. Table 2 details the concrete mixtures used in the study. The cement used in this study was ordinary Portland type produced from Tasluja - Iraqi origin bazian, while the sand (fine aggregates) and gravel (coarse aggregates) were supplied from Al-Ukhaidir region, south-west of Baghdad and Al-Nebai region, north-west of Baghdad, respectively. All components are complying with the Iraqi standard of 1984. Drinking tap water has been employed to prepare the concrete mixture. Tables 3-5 show the properties of all components used.

Table 2. Details of the concrete mixtures used in this study

Type of mixture	Cement, Kg/m^3	Sand, Kg/m^3	Gravel, Kg/m^3	W/C, ratio	Waste Tea Leaves, (WTL)
Reference	300	600	1200	0.45	-
Mixture 1	300	600	1200	0.45	1.8% of WTL
Mixture 2	300	600	1200	0.45	0.1-2.8% Ni^{+2}-WTL

Table 3. Properties of Tasluja-Bazian cement used in this study (Rajaa et al., 2023)

Component	Cement Used	Specification Limits, According to Iraqi Specification (No. 5 of 1984)
Calcium oxide (CaO)	62.42	60 – 69
Silica (SiO_2)	21.30	18 – 24
Alumina (Al_2O_3)	5.52	4 – 8
Ferric Oxide (Fe_2O_3)	4.25	2 – 4
Magnesium Oxide (MgO)	2.53	2 – 5

Potassium Oxide (K_2O)	0.72	
Sodium Oxide (Na_2O)	0.53	
Sulfide Oxide (SO_3)	0.83	0.3 – 2.7
Free Lime (CaO_F)	1.36	0 – 2
Insoluble Residue (I.R.)	0.54	
Loss On Ignition (L.O.I)	0.5	0.1 – 0.5
Lime Saturation Factor (L.S.F)	0.8935	0.66 – 1.02
Tri-Calcium Silicate (C_3S)	49.00	45 – 65
Di-Calcium Silicate (C_2S)	23.88	10 – 25
Tri-Calcium Aluminate (C_3A)	7.57	7 – 12
Tetra-Calcium Aluminate Ferrite (C_4AF)	13.00	11 – 15

Table 4. Properties of sand used in this study (Rajaa et al., 2023)

Sieve size, (mm)	Passing by weight %	Iraqi specification limit (No. 45 of 1984), area 2
10	100	100
4.75	94	90 - 100
2.36	84	75 – 100
1.18	78	55 – 90
0.6	56	35- 59
0.3	27	8 – 30
0.15	3	0 – 10
Soft materials passing through sieve No. 200	0	< 5%

Table 5. Properties of gravel used in this study (Rajaa et al., 2023)

Sieve size, (mm)	Passing by weight %	Iraqi specification limit (No. 45 of 1984), area 2
37.5	100	100
20	99	95 – 100
10	42	30 – 60
5	2	0 – 10
Materials passing through sieve No. 75 µm	0	<3%

Testing Method (Compressive Strength Test): The compressive strength of concrete used for structural works is usually expressed by the unit of force (stress) required to cause rupture in (Newton) divided by the area unit in (mm^2). To determine the compressive strength of the concrete mixture, a cube sample of 15×15×15 cm or a cylindrical sample, with a height equal to twice the diameter. The treatment is performed by wet method for 7 days and 28 days according to the Iraqi specification at lab temperature. The sample is then loaded by putting it in a standard concrete compressive strength testing machine with a specific velocity until it is ruptured. The loading was achieved usually within 2-3 minutes. In this study, the compressive strength test was carried out according to British standard specification (B.S. 1881: part 116) for 7- and 28-day concrete cubes prepared using a compressive strength testing machine (WeKob) of 2500kN capacity and

$15 MN.m^{-2}.min^{-1}$ until the rupture of the sample starts. Cubes of reference concrete mixture and additive concrete mixtures for every age (i.e., 7 and 28 days) were tested to identify the best compressive strength. The dependent compressive strength was the average of three cubes for each test in each age.

Obvious from Figure (12) is the enhanced effect of nickel loaded waste tea leaves on the concrete mixture behavior. Using the Ni^{2+}-loaded WTL, the compressive strength of cubes increased gradually from 27.41 $kN.mm^{-2}$ at a ratio of 0.1 to a maximum value of 43.17 $kN.mm^{-2}$ at a ratio of 1.8, while at the same optimum ratio (1.8%) the compressive strength was found to be 30.43 N/mm^2 at 7 days and 33.29 N/mm^2 at 28 days, using WTL alone (free from Ni^{2+} ions). The increase in the compressive strength of concrete mixture due to the addition of the waste tea leaves loaded with nickel ions may be due to the increased strength of adhesion between the loaded waste tea leaves and the concrete cement. The adhesion depends on some physical and chemical properties resulting from the structure of the waste tea leaves. Moreover, the compressive strength for this case was increased due to the existence of nickel ions on the surface of waste tea leaves. The presence of nickel (which is a high-density element) by a quantity that cannot be ignored certainly increases the compressive strength of concrete due to nickel penetration into the pores and voids of the concrete mixture, because of being loaded by waste tea leaves. Beyond the loaded waste tea leaves ratio of 1.8, the compressive strength started to decrease gradually until the complete failure of samples at a ratio of 2.8. This result can be explained by the same reasons illustrated in case of the addition of waste tea leaves alone.

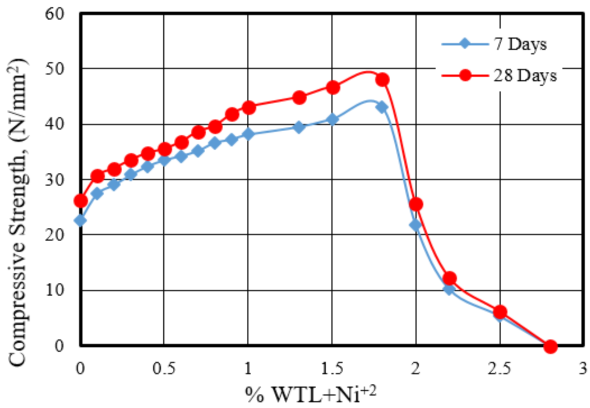

Figure (12): Effect of Ni^{+2} – waste tea leaves added to concrete

Comparison between This Study and Other Studies

While the current manuscript is concerned with studying the ability of waste tea leaves to remove nickel ions from contaminated aqueous solutions, various papers delved into the possibility of removing nickel ions from contaminated water using different adsorbents. Table 6 presents a comparative analysis of the findings between this study and previous studies conducted by different researchers.

Table 6. Summary of nickel adsorption using different adsorbents

Adsorbent	Operating Conditions			Capacity, (mg/g)	Reference
	pH	Dose, (g/l)	Time, (min)		
Coir pith	7		10	38.9	Ewecharoen et al., 2008
Pomegranate peel	6	10	4200	52	Bhatnagar and Minocha, 2010
Treated rice brane	6		30	153.6	Zafar et al., 2015
Coconut waste	5	20	120	3.77	Saleem et al., 2016
Peanut hull ash	6.2	3	140		Wu et al., 2011
Montmorillonite	6	1	60	46	Barati et al., 2013
Purified Muti-Walled Carbon Nano-Tubes	6	0.4	40		Adolph et al., 2012
Activated carbon	7	3	115	11.9	Maddodi et al., 2020
Banana peels	8	4.5	120		Olufemi and Eniodunmo, 2018
Watermelon rind	5	0.05	30	35.3	Lakshmipathy and Sarada, 2013
Waste Tea Leaves	6	5.5	120	0.567	This study

CONCLUSIONS

Discarded in millions of tons annually, waste tea leaves could be a sustainable source for water treatment. Results reveal a surface area of nearly 12.251 m^2/g, suggesting a significant potential for recovering nickel metal, a carcinogenic substance, from aqueous solutions under various operational conditions. The removal efficiency directly correlates with key factors; acidity, waste tea leaves dose, contact time, and agitation speed; while inversely relating to temperature, and the initial concentration of nickel. Under specific conditions of pH 6, a contact time of 120 minutes, a temperature of 20°C, 5.5 g of waste tea leaves, nickel initial concentration of 42 ppm, and agitation speed of 350 rpm, the highest removal percentage, reaching 74.301%, is achieved. Morphological investigations, including BET, FTIR, pH_{pzc} and FESEM analyses on both virgin and loaded waste tea leaves, illustrate the impact of nickel adsorption on pore closure. BET analysis indicates that the adsorption process occupies 87.5% of the surface area before treatment, and FTIR results highlight the disappearance and shift of numerous peaks, emphasizing the significant role of adsorption. FESEM observations elucidate the closure of pores within waste tea leaves due to nickel adsorption. The point of zero charge experiments shows that 6.979 is the pH_{pzc} of waste tea leaves used in this study. Finally, as a method to benefit from the residues, the loaded waste tea leaves double the compressive strength of concrete mixtures.

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