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Review

# Role of Black Tea in the Advancement of Nanotechnology: A Critical Review

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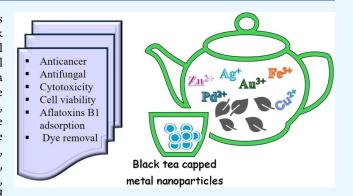
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ABSTRACT: Black tea (BTE), a well-used beverage, has immense applications in nanotechnology. The extract of black tea can perform as a reducing and capping agent for forming metal and metal oxide nanoparticles (NPs). Though many metal nanoparticles are reported to be formed employing black tea extract, Ag- and Fe-based nanoparticles are mostly found in the literature. Among the different constituents of BTE extract, catechin and epigallocatechin gallate are responsible for the passivation of the nanoparticles due to the oxidation of the polyphenol moiety. Such nanoparticles, being capped with BTE, exhibit direct and indirect applications, including anticancer, antifungal, cytotoxicity, cell viability, Aflatoxins B1 adsorption, and dye removal applications. BTE is a natural reducing and



Article Recommendations

capping agent that produces zero-valent metal nanoparticles. However, ZnO and CuO nanoparticles are also found in the literature due to subsequent oxidation of the zero-valent metalized surface. We included the synthesis, applications, and mechanistic details of nanoparticles passivated with BTE. Moreover, we summarized the effect of components in nanotechnological applications. As BTE is nontoxic, its involvement makes the process green and warrants a detailed review for the young researcher in nanotechnology. No review article is available regarding the nanometric application of BTE, justifying our present Review's novelty and originality.

# MOTIVATION

Though black tea (BTE) is mostly used in beverages and is cheap, its nanometric use cannot be neglected. Currently, the role of BTE is being published by leaps and bounds. Green tea is also being researched on a similar ground. A review article was found on brewing nanochemistry with green tea by Gupta et al. Luczaj and E. Skrzydlewska reviewed the antioxidative properties of black tea.<sup>2</sup> Li et al. reviewed the chemical analysis and stability of BTE.3 However, no review was available on BTE's usage in nanotechnology. We tried to fill the gap with the present review article.

# 1. INTRODUCTION

Nanotechnology studies and advances materials at the atomic, molecular, or macromolecular levels. NPs are particles with a minimum dimension of approximately 100 nm and are regarded as the fundamental components of nanotechnology. 4-6 For thousands of years, various industries and humankind have employed particles within these size ranges; however, there has been an increase in their use because of the capacity to produce and fabricate these materials. Nanoscale materials are employed in various applications, including electronic, magnetic, and optoelectronic, as well as biomedical, pharmaceutical, cosmetic, energy, environmental, catalytic, and

materials applications. The potential of this technology has resulted in a global rise in investment in nanoscience and development.7

NPs have been implemented in numerous scientific applications. Typically, the nanoparticles are manufactured in large quantities on a commercial scale and are unsealed, with a larger dimension. When these nanoparticles are discharged in the form of massive aggregates, they become environmentally hazardous. Consequently, the selection of appropriate capping constituents is crucial for the stabilization of colloidal solutions and their absorption into living cells and the ambient environment.9,10

Consequently, the selection of appropriate capping moieties is crucial for the stabilization of colloidal solutions and their absorption into living cells and the environment. Capping nanoparticles with biocompatible surfactants results in modifications to their surface chemistry and size distribution.

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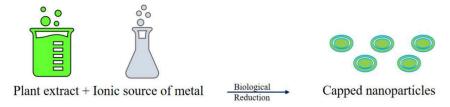


Figure 1. Schematic representation of the green synthesis of nanoparticles.

To facilitate their utilization within the living system, capping agents must be biodegradable, biocompatible, nontoxic, well-dispersed, and biosoluble. 11

The researchers are highly interested in the assessment of the prospective therapeutic potential and environmental impact of nanoparticles. Surface sealing improves the biological properties of nanoparticles. Capping agents are novel therapeutic agents that exhibit clinical significance when they are combined with biocompatible nanoparticles to which they have been affixed. The nanocomposite's ultimate stability is achieved through the steric hindrance that results from the covalent bonding between the chains of capping ligands and the nanoparticles' surface. The nanoscale results in an increase in the percentage of atoms on the surface, which is further augmented by capping. The evaluation of the potential therapeutic potential and environmental impact of NPs is of great interest to the researchers.

Surface coating improves the biological properties of NPs. Capping agents are novel therapeutic agents that exhibit clinical significance when they are combined with biocompatible nanoparticles to which they have been connected. The nanocomposite's ultimate stability is achieved through the steric hindrance that results from the covalent bonding between the chains of capping ligands and the NPs surface. The nanoscale increases the percentage of atoms on the surface, which is further augmented by capping. Additionally, the agglomeration of nanoparticles is reduced for an extended period through the use of suitable coating agents. 12,9

Fabricating nanoparticles by employing plant compounds as prospective capping and reducing agents is frequently referred to as "green synthesis". The properties of the formulated nanoparticles can be influenced by the compounds from the leaves, fruit, roots, and seeds of the plants, which can optimize their functionality for biomedical applications (Figure 1). 9,14

Plant extracts have diverse applications in nanotechnology. Sathiyabama et al.<sup>16</sup> used tea extract and its antibacterial properties against economically significant rice phytopathogens, synthesizing chitosan nanoparticles. Baldemir et al. 17 synthesized nanoflowers with green tea (Camellia sinensis (L.) Kuntze) extract and its main constituents to boost antibacterial activity. Kalayci et al. 18 formed flower-shaped nano biocatalysts using green tea and investigated their peroxidase-mimicking activity on the polymerization of phenol derivatives. For cancer treatment, Chung et al. 19 developed self-assembled micellar nano complexes using protein medications and green tea derivatives. Wirwis et al.20 showed the green-chemical synthesis of Ag nanoparticles employing tea leaf extract for enhanced physicochemical activities. Iron oxide nanostructures, that were phytosynthesized using an aqueous extract of Saccharum arundinaceum (hardy sugar cane), were also described, along with their cytotoxic, antiglycation, and characterization properties.<sup>21</sup>

BTE extract serves as a capping agent. BTE is selected as a surfactant/capping agent due to its substantial polyphenolic content, primarily composed of theaflavins and thearubigins. Due to their complex and steric characteristics, these polyphenols serve as stabilizing or capping agents. The consortium effect of the tea extract impedes particle proliferation. Theaflavins are present in BTE extract, a surfactant. Theaflavins are essentially antioxidant polyphenols. During the production of BTE, theaflavins are produced through the polymerization of catechin during the fermentation or semifermentation procedure (Figure 2). 22,23



**Figure 2.** Preparation of BTE-capped gold nanoparticles with a microscopic SEM image. Reprinted with permission from ref 15. Copyright 2009 The Royal Society of Chemistry.

In this review article, we focused on the chemistry of BTE, the components of BTE, BTE-NPs, their applications, and the fate of black tea-capped nanoparticles.

### 2. TYPES OF TEA

The domesticated evergreen plant known as tea (*Camellia sinensis* L.) originated in China. Then it went to India, Japan, Europe, and Russia before reaching the New World in the late 17th century. Though they are all produced from the same plant species—C. sinensis L.—green, oolong, and black teas differ in terms of flavor, organoleptic taste, and chemical content.<sup>24</sup>

The leaves of the tropical evergreen shrub Camellia sinensis are used to make tea. With a per capita intake of around 120 mL day-1, tea is the second most prevalent brew due to its appealing and unique color, flavor, fragrance, taste, and health advantages. Three forms of tea exist: unfermented (green tea), semifermented (oolong tea), and fermented (black tea). While green tea is more popular in Asia, black tea is more widely drunk in North America, Europe, and North Africa. Taiwan and China are the two main places where oolong tea is produced. Every year, over three billion kg of tea is produced and consumed. Over the globe, around 76–78% of BTE, 20–22% of green tea, and less than 2% of oolong tea are formed and drunk<sup>25</sup> (Table 1).

# 3. CHOICE OF BLACK TEA

BTE is considered a common beverage and an antioxidant agent accessible in everyday life. It is consumed worldwide. The most common traditional brew in the world is tea, and

Table 1. Composition of Different Types of Tea

type of tea	production percentage	consumption percentage	primary regions
BTE	76-78%	76-78%	North America, Europe, North Africa
green tea	20-22%	20-22%	Asia
oolong tea	<k2%< td=""><td>&lt;2%</td><td>Taiwan, China</td></k2%<>	<2%	Taiwan, China

BTE has a variety of biological impacts on different living things. Its capacity to scavenge free radicals and chelate metals makes it an excellent antioxidant.<sup>26</sup> The biological activity of green tea has been extensively investigated; however, BTE comprises approximately 80% of the 2.5 million metric tons of tea. Consequently, in recent years, BTE has been the subject of extensive research, particularly emphasizing its impact on human health.<sup>2</sup>

#### 4. ORIGIN OF BLACK TEA AND ITS POLYPHENOLS

Green tea fermentation is traditionally thought of as the step in the production of BTE. But it is not the same as the microbial fermentation used to make vinegar, alcoholic drinks, and other products. Two steps are involved in the production of BTE polyphenols: oxidation and polymerization.<sup>27,3</sup>

Under the enzymatic catalysis of polyphenol oxidase (PPO, EC1.14.18.1) or peroxidase (POD, EC1.11.1.7), green tea catechins, such as epigallocatechin (EGC) and epigallocatechin gallate (EGCG), are partly oxidized to quinones in the first reaction step, as shown in Figure 3 There are naturally occurring PPO and POD in fresh tea leaves, fruits, and vegetables.<sup>3</sup>

The second stage, which is frequently referred to as polymerization, is the process of converting the resulting gallo-catechin quinones into catechin quinones through a nucleophilic addition reaction. Additional oxidation with oxygen or hydrogen peroxide, the removal of  $CO_2$ , and rearrangement are then involved in synthesizing benzotropolone, the main component of BTE polyphenols. It is reasonable to assume that the production of a stable aromatic bicyclic ring known as benzotropolone, as well as the removal of small molecules ( $CO_2$  and  $H_2O$ ), are what propel this enzymecatalyzed activity (Figure 3).<sup>3</sup>

# 5. THE GRADING OF BLACK TEA

The descriptive sensory analysis, aroma-active compounds (volatile compounds), and taste-active compounds (sugar, organic acid, and free amino acid components) of seven grades of BTE [high-quality BTE (grades 1 to 3) and low-quality BTE (grades 4 to 7)] processed by ÇAYKUR Tea Processing Plant (Rize, Turkey) were compared.<sup>28</sup>

In Turkey, the ÇAYKUR Tea Processing Plant processes over 50% of the black tea market. The plant processes BTE according to its own seven distinct categories, which include high-quality tea (grades 1-3) and low-quality tea (grades 4-7).] (Table 2). The low-quality tea groups are marketed after being blended with high-quality group categories to the demands.<sup>28</sup>

# 6. THE COMPONENT OF BTE

The composition of tea is contingent upon the maturity of the leaf, which is determined by its location on the harvested stalk, along with climate, season, horticultural practices, and

variety.<sup>29</sup> About the dried substance mass, fresh tea leaves contain an average of 36% polyphenolic compounds, 25% carbohydrates, 15% proteins, 6.5% lignin, 5% ash, 4% amino acids, 2% lipids, 1.5% organic acids, 0.5% chlorophyll, and carotenoids and volatile substances that account for <0.1%. Polyphenols are the most prevalent compounds, with catechins being the most prevalent.<sup>29</sup> Epigallocatechin gallate (EGCG), epigallocatechin (EGC), epicatechin gallate (ECG), and epicatechin (EC) are the most significant catechins<sup>2415</sup>. Gallocatechin, epigallocatechin digallate, 3-methylepicatechin gallate, catechin gallate, and gallocatechin gallate are also in lesser quantities. The concentration of catechins in tea leaves is contingent upon their age. It is typically higher in the leaves used for black tea production than in those used for green tea manufacture. 29,2 The composition of BTE is contingent upon the technological process of its manufacture. It is challenging to establish a certain composition for BTE beverages, as they are subject to variation depending on the preparation method. The mean percentages of solid extract components in BTE beverages are approximate as follows: catechins (10-12%), theaflavins (3-6%), thearubigins (12-18%), flavonols (6-8%), phenolic acids and depsides (10-12%), amino acids (13-15%), methylxanthines (8-11%), carbohydrates (15%), proteins (1%), mineral matter (10%), and volatiles (0.1%) (Figure 4).<sup>2</sup>

# 7. SYNTHESIS OF BLACK TEA-CAPPED NANOPARTICLES

There are numerous non-nanometric applications of BTE in our practical life. The cosmetics market is significantly influenced by the centuries-old tradition of use of the tea plant and its extracts. Nille et al. Summarized valorization of tea waste for multifaceted applications BTE is equally important in nanotechnology. Employing the reducing behavior of polyphenols presenting in black tea, NPS are synthesized for myriad applications. Black tea extract is often used simultaneously as a reducing and stabilizer in the context of various NPs formation (Table 3).

Different brands and countries of BTE were used for black tea-capped nanoparticle synthesis; black tea (Loyd, Mokate, and Poland)<sup>41</sup> Lahijan, Iran<sup>47</sup> local health food store<sup>42</sup>

Two steps are involved in BTE-capped nanoparticle synthesis. The first is the extraction of BTE and the Preparation of NPs by tea extract. Several conditions were used to extract BTE extract. The ultrasonic-assisted exaction method (Ultrasonic frequency: 25 kHz, input power: 150W), solvent: solid ratio: 1:3 m/m, extraction rate: 15% were used by Both et al.<sup>50</sup> Applying heat causes plant cells to become more permeable by rupturing them. It also prevents polyphenols from interacting with lipoproteins, increasing the solubility of polyphenols in water. The greatest amount of polyphenol content may be extracted using heat extraction.<sup>39</sup>

Synthesis of nanoparticles from BTE can be performed in various conditions, such as microwave-assisted, sonochemical, sol–gel, electrolytic deposition, mechanical stirrer, room temperature, etc. Using tea as an electron donor and stabilizer, Nune et al. formed four distinct ways for synthesizing gold nanoparticles at various reaction temperatures, either with or without the inclusion of gum Arabic. <sup>15</sup> BTE extract is used to reduce gold ions into gold nanoparticles. This starts the reduction process, which turns Au<sup>3+</sup> ions into Au<sup>0</sup> nanoparticles. <sup>32</sup> Gol et al., <sup>35</sup> using AgNO<sub>3</sub> salt and *Camellia sinensis* extract in a reduction process, produced silver nanoparticles

i) Nucleophilic reaction ii) Oxidation, 
$$O_2$$
 or  $H_2O_2$  iii) Elimination,  $-Co_2$ 

HO

OH

Theaflavins

 $R_1 = R_1' = H$ ,  $TF1$ ;

 $R_1 = galloyl$ ,  $R_1' = H$ ,  $TF2b$ ;

 $R_1 = H$ ,  $R_1' = galloyl$ ,  $TF2b$ ;

 $R_1 = R_1' = galloyl$ ,  $TF3$ 

Figure 3. Flavin production pathway catalyzed by enzymes.

sonochemically. After that, the produced nanoparticles were dried and cleaned for further usage. BTE might be used to produce Ag nanoparticles in an alkaline environment. Controlled reduction and formation of nanoparticles were made possible by the partial addition of AgNO<sub>3</sub>.

Silver nanoparticles using BTE and green tea infusions, taking advantage of the reducing properties of the tea extracts facilitate the formation of nanoparticles. Adjusting the pH to 10 helped optimize the reduction process, resulting in the efficient synthesis of AgNPs.<sup>37</sup> Green tea and BTE leaf extracts were used to form silver nanoparticles by combining the

Table 2. Seven Grades of BTE

quality	grade	acronmy	description		
high-quality BTE	grade 1	OF	orange fannings		
	grade 2	BOP-1	broken orange pekoe-1		
	grade 3	OP	orange pekoe		
low-quality BTE	grade 4	F	fannings		
	grade 5	BOP-2	broken orange pekoe-2		
	grade 6	BP	broken pekoe		
	grade 7	FD	fine dust		

extracts with silver nitrate aqueous solution in a 9:1 volume ratio. Solution was used to produce copper nanoparticles from black tea and green tea. Each tea leaf extract was combined with 1 mmol/L of CuSO<sub>4</sub> in a 4:1 volume ratio. Leaf extracts from BTE and green tea were used to produce iron nanoparticles. Each tea leaf extract received a separate addition of 1 mmol/L of FeCl<sub>3</sub> solution at a volume ratio of 1:1. The reduction of Fe<sup>2+</sup> ions was demonstrated with black precipitation. The extracts of BTE, green, and oolong teas are often used to produce iron nanoparticles Synthesizing nanoparticles of zero-valent iron modified with BTE extract was well-reported. The Lahijan Tea Research Centre in Lahijan, Iran is the source of the black tea leaves utilized in this synthesis. Lebaschi et al. synthesized palladium nanoparticles from BTE leaf.

the biogenic synthesis method, BTE extract-silver/poly(vinyl alcohol) nanocomposite films were manufactured (Figure 5).<sup>51</sup>

Important details on the stability of nanoparticle dispersion are provided by the zeta potential (z). The long-term stability of the nanoparticulate dispersion may be predicted based on the magnitude of the observed zeta potential, which indicates the presence of repulsive forces. The equilibrium of the repulsive and attractive forces between nanoparticles as they get closer to one another determines how stable the dispersion of nanoparticles is. The dispersion will be stable if every particle repels every other particle. Aggregation occurs when there is little to no repulsion between the particles. Nune et al. 15 looked at how stable BTE AuNPs were in phosphate buffer solutions with pH values of 5, 7, and 9. The plasmon wavelengths, in the above-mentioned conditions, exhibited slight variations of 1 to 5 nm. These in vitro stability experiments' findings demonstrated the exceptional in vitro stability of BTE AuNPs in biological fluids at physiological pH. Table 1 summarizes the zeta potential of BTE-capped nanoparticles. Das et al.<sup>32</sup> tabulated the time-dependent values of hydrodynamic diameter (nm), z, and polydispersity index (PDI) of BTE-AuNPs in acidic buffer, PBS, as well as basic buffer (Table 4). The consistent size distribution of NPs was shown by the PDI. The reduced PDI value was due to uniform size and monodispersity. Some polydispersity was observed in acidic and PBS buffers, and a slight rise in the basic buffer.

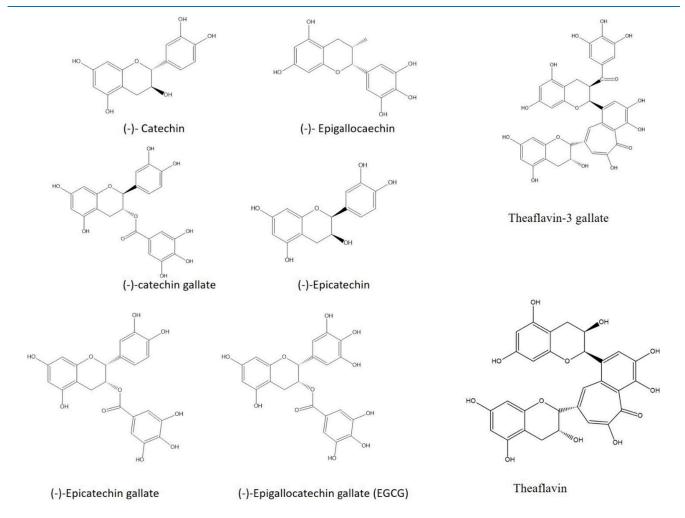
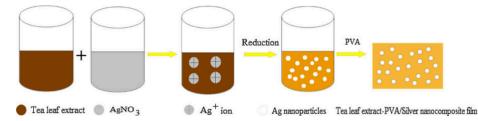


Figure 4. Composition of various phytochemicals in black tea leaves.

Table 3. Synthesis and Properties of Metal Nanoparticles Passivated with BTE

synthesized				
NPs	experimental parameter	size (nm)	shape	zeta potential
AuNPs <sup>32</sup>	BTE (150 mg in 15 mL water at 20 $^{\circ}\text{C}$ and filtered) + 15 mL of 1 mM HAuCl $_{4}\textsc{;}$ stirred for 15 min	Table 4	circular and spherical	Table 4
AuNPs <sup>33</sup>	BTE (180 g boiled in 400 mL water and filtered) + 75 $\mu L$ of 0.01 M HAuCl $_4$ ; stirred for 30 min at 40 $^{\circ} C$		nanoprisms, nanorods, and nanotrapezoids	
AuNPs <sup>34</sup>	BTE (180 g boiled in 1.2 mL water and filtered) + 10 mL of 1 mM HAuCl $_4$ ; constant magnetic stirred	38	spherical	
AgNPs <sup>33</sup>	BTE (180 g boiled in 400 mL water and filtered) + 75 $\mu L$ of 0.01 M AgNO3; stirred for 30 min at 40 $^{\circ} C$	20	spheroidal, nanoprisms, nanorods and small aggregates	
AgNPs <sup>35</sup>	20 mL BTE (5.0 g mixed with 100 mL 70% ethanol) + 15 mg of AgNO $_{3}\!;$ held in the ultrasonic type sonication for 2 days	$14.9 \pm 1.4$	spherical	
AgNPs <sup>36</sup>	80 mL BTE (1.5 g in 250 mL water heated for 20 min and filtered) + 5 mL AgNO $_3$ (54 mg/200 mL water); stirring was maintained at the minimum speed; pH 10	not detectable		
AgNPs <sup>37</sup>	200 BTE (1.5 g in 250 mL water heated for 20 min, filtered and diluted to 500 mL) + 67 mg AgNO $_3$ ; stirring was maintained at the minimum speed; pH 10	30-50	spherical	-19 mV
AgNPs <sup>38</sup>	BTE (10 g in 100 mL water heated for 20 min, at 80 $^{\circ}\text{C}$ temperature, filtered): $\text{AgNO}_3$ (1 mmol/L) = 1:9; stirred at room temperature	14-20	spherical	
AgNPs <sup>39</sup>	10% BTE (10 g in 100 mL stirred for 4 h at 40 °C, filtered) + 10 mM AgNO $_{3};150$ min; 700 rpm	50-120	aggregated	22.7 mV
AgNPs <sup>40</sup>	BTE (0.6–4 g in 100 mL stirred for 25 min at 60 –70 °C, filtered) + 1–5 mM AgNO3; 12 h			
AgNPs <sup>22</sup>	2.5% BTE (0.3433 g in 20 mL of boiling water for 30 s, filtered) + 0.005N AgNO_3); electrolysis	2-25	spherical	
AgNPs <sup>41</sup>	25 mL BTE (10 g in 100 mL of water, 60 °C for 15 min, filtered) + 750 mL, 10 mM AgNO <sub>3</sub> ); <50 °C; 2 h	30-60	spherical and triangular	
CuNPs <sup>38</sup>	BTE (10 g in 100 mL water heated for 20 min, at 80 $^{\circ}$ C temperature, filtered): CuSO <sub>4</sub> (1 mmol/L) = 1:4; stirred at room temperature	32-40	spherical	
CuO NPs <sup>42</sup>	30 mL BTE (2 g in 100 mL of water, boiled for 30 min, filtered) + 2 g of Cu(NO $_3)_2\cdot$ 3H $_2O$ in 10 mL; stirred for 12 h	35-65	spherical shape	
CuO NPs <sup>43</sup>	50 mL BTE (1 g in 100 mL of water, at 80 $^{\circ}C$ temperature filtered) + 50 mL 0.1 M Cu(CH <sub>3</sub> COO) <sub>2</sub> ·H <sub>2</sub> O; stirred for at 60 $^{\circ}C$ for 1 h	10-12	spherical	
FeNPs <sup>38</sup>	BTE (10 g in 100 mL water heated for 20 min, at 80 $^{\circ}C$ temperature, filtered): FeCl $_3$ (1 mmol/L) = 1:1; stirred at room temperature	46-60	spherical	
FeNPs <sup>44</sup>	BTE (60.0 g/L, heated at 80 °C temperature, for 1 h, filtered) = (0.10 mol/L FeSO <sub>4</sub> ); 1:2	40-50	spherical	
FeNPs <sup>45</sup>	BTE (10 g in 100 mL water heated for 10 min, at 80 °C temperature, filtered): FeCl $_3$ (8.11 g in 500 mL) = 1:1; stirred at room temperature	200		
FeNPs <sup>46</sup>	BTE (20.0 g in 200 mL water heated for 30 min, at 85 $^{\circ}\text{C}$ temperature, filtered) + 0.10 M FeCl $_3$	70	irregular square particles	-22.05 mV
PdNPs <sup>47</sup>	10 mL BTE (10 g in 100 mL water heated for 5 min, filtered) + 100 mL of 1 mM PdCl $_2\!\!$ ; refluxed at 100 °C for 1 h	5-8	spherical	
ZnO NPs <sup>48</sup>	30 mL BTE (2 g in 100 mL water heated for 30 min, filtered) + 2 g of Zn(NO $_3$ ) $_2$ · 6H $_2$ O was dissolved in 10 mL; stirred at 75 °C for 12 h	32	hexagonal	
ZnO NPs <sup>49</sup>	30 mL BTE (2 g in 100 mL water heated for 30 min, at 70 °C temperature filtered) + 2 g of $Zn(NO_3)_2$ ·6H <sub>2</sub> O was dissolved in 10 mL; stirred at 75 °C for 12 h	80-430	hexagonal wurtzite	



**Figure 5.** Diagrammatic representation of the synthesis of the BTE leaf extracts—Ag/PVA nanocomposite film. Reprinted with permission from ref 51. Copyright 2012 Elsevier.

**7.1. Formation Mechanism.** Tea extract is a phytochemical with different types of polyphenols. The composition of BTE was discussed in section 5.

7.1.1. The Component of BTE. Oxidation of phenols to quinone form is associated with reducing metal ions to metal nanoparticles. For example, gallic acid is an important component of BTE. It causes the stability and reduction of silver nitrate to silver nanoparticles. Gallic acid's phenolic

groups combine with  $Ag^+$  ions to produce intermediate complexes, according to Edison and Sethuraman. These compounds oxidize gallic acid to quinone form and reduce  $Ag^+$  to zero-valent silver nanoparticles (Figure 6).  $^{53,54}$ 

The chemical components found in the extract of the leaves of the *Piper pedicellatum* plant—gallic acid, protocatechuic acid, courmaric acid, and catechin—were shown to have reduced and capping properties, as reported by Ismail et al. 55

Table 4. Time-Dependent Values of Hydrodynamic Diameter (nm), Zeta Potential, and Polydispersity Index (PDI) of BTE-AuNPs in Acidic Buffer, PBS, and Basic Buffer

AuNPs in different pH	hydrodynamic diameter (nm) on 12 h	hydrodynamic diameter (nm) on 24 h	hydrodynamic diameter (nm) on 36 h	zeta potential (mV) on 12 h	zeta potential (mV) on 24 h	zeta potential (mV) on 36 h	PDI for 12 h	PDI for 24 h	PDI for 36 h
BTE-AuNPs in acidic buffer (pH 5.5)	56.1 ± 1.1	$76.3 \pm 1.8$	$98.6 \pm 2.1$	$-12.5 \pm 0.1$	$-20.9 \pm 0.1$	$-25.2 \pm 1.1$	0.290	0.330	0.360
BTE-AuNPs in PBS (pH 7.4)	$33.4 \pm 0.8$	$55.6 \pm 1.2$	$75.6 \pm 1.5$	$-6.56 \pm 0.01$	$-9.45 \pm 0.09$	$-13.5 \pm 0.3$	0.310	0.340	0.380
BTE-AuNPs in basic buffer (pH 9.5)	$66.2 \pm 1.2$	$92.5 \pm 2.0$	$112.5 \pm 2.5$	$-15.3 \pm 0.4$	$-18.7 \pm 0.6$	$-29.6 \pm 1.5$	0.350	0.390	0.440

<sup>&</sup>lt;sup>a</sup>Reprinted with permission from ref 32. Copyright 2022 The Royal Society of Chemistry.

Figure 6. Mechanism of the Formation of Silver Nanoparticles from Ionic Silver Using Gallic Acid

$$(-OH \ catechin) \qquad (-OH \ gallic \ acid) \qquad (Radical) \qquad (Radical)$$

$$2 \ Radical \qquad + \ 2Ag^{+} \qquad \longrightarrow \qquad Oxidized \ catechin \ + \ Oxidized \ gallic \ acid \ + \ 2Ag^{0} \ (Nucleation)$$

$$Ag^{0} \qquad + Ag^{+} \qquad Fast \qquad Ag_{2}^{+} \quad (growth)$$

$$Ag_{2}^{+} \qquad + Ag_{2}^{+} \qquad Fast \qquad Ag_{3}^{2+} \quad (growth)$$

$$(Ag_{4}^{2+})_{n} \ (catechin)_{n2} \qquad \longrightarrow \qquad Yellow \ brown \ colour \ Ag \ NPs \ solution$$

Gallic acid radical

Figure 7. A plausible mechanism to show how the hydroxyl groups of gallic acid and catechin in the extract of *Piper pedicellatum* leaves reduce Aginore

Additionally, they put out a speculative mechanism for how the hydroxyl groups of gallic acid and the catechin in the extract of *Piper pedicellatum* leaves reduce Ag<sup>+</sup> ions. BTE has similar components, and consequently, similar mechanisms are applicable (Figure 7).

Gallic acid

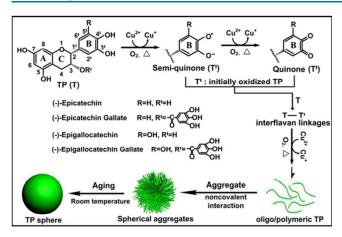
On the other hand, Chen et al. reported Cu<sup>2+</sup> and temperature-mediated oxidative coupling mechanisms for synthesizing green luminous, monodisperse, smooth, porous, and hollow spheres of tea polyphenols with ease. Their method only used temperature-mediated oxidative coupling assembly of green tea polyphenols (TP) and copper (II, Cu<sup>2+</sup>) to create biocompatible hollow, porous, and smooth spheres. The catechol and pyrogallol groups of (epi)catechins and (epi)-(gallo)catechins gallates (T) were first transformed into corresponding highly reactive semiquinones and quinones under heating conditions and ambient environment. Quinone was mostly involved in the following coupling production of interflavan linked dehydrodicatechins (T-T') by nucleophilic addition, although a semiquinone radical might also play a role in the coupling process. The redox cycling of the Cu<sup>2+</sup>/Cu<sup>+</sup>

pair and the coordination of Cu to catechins facilitated this activity.

3-4- didehydroshikimate

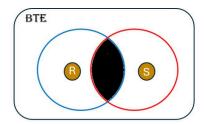
Cu(II) accelerated the oxidation coupling and acted as catalysts in the oxidation polymerization of aromatic compounds and in plant browning via copper-containing glycoproteins (Figure 8). 56,57

**7.2.** Epigallocatechin Gallate as a Reducing Agent. Using tea's phytochemicals, such as theaflavins, thearubigins, and catechins, is a noteworthy advancement in the synthesis and stabilization of gold nanoparticles. Strong reducing qualities are possessed by phytochemicals such as catechin and epigallocatechin gallate (EGCG). Through the donation of electrons during the reduction reaction, they aid in the transformation of gold ions in solution into gold nanoparticles. These phytochemicals also function critically in stabilizing gold nanoparticles once they develop. They encircle the nanoparticles in a protective layer that keeps them from clumping or aggregating. The long-term stability of the nanoparticles in aquatic conditions is guaranteed by this coating. Furthermore, using gum Arabic as a stabilizing agent improves the nanoparticles' stability even further. Gum Arabic functions as



**Figure 8.** Schematic illustration of temperature and Cu<sup>2+</sup>- mediated oxidative assembly of TP. Reprinted with permission from ref 57. Copyright 2013 American Chemical Society.

a surfactant, assisting in preserving.<sup>15</sup> The reducing and capping qualities of BTE triggered the formation of the *Camellia sinensis* silver nanoparticles (Figure 9).<sup>36</sup>



- Possible Role as Reducing Agents:
  Catechins (Catechin, Epicatechin, Epicatechin gallate,
  Catechin gallate, Epigallocatechin, Epigallocatechin gallate), Theaflavins.
- S Possible Role as Stabilizing Agents:
  Catechins (Catechin, Epicatechin, Epicatechin gallate,
  Catechin gallate, Epigallocatechin, Epigallocatechin gallate), Theaflavins and
  Thearubigins



**Figure 9.** Venn diagram illustrating the potential function of phytochemicals in BTE for generating and stabilizing AuNPs.

Use of the chemical epigallocatechin gallate (EGCG) found in BTE extract (electron donor and stabilizer) during the formation of copper oxide NPs. (Figure 9) depicts this process and shows how polyphenols like EGCG contribute to the quick production and stabilization of metallic nanoparticles in an aqueous media. 42

- **7.3. Complex Formation.** Two of the copper ion's sp orbitals can be occupied by the lone pair electrons found in the polar groups of EGCG, creating a complex combination. Because of this interaction, EGCG can cap the copper ions, which makes it easier to create Cu<sup>0</sup> nanoparticles inside nanoscopic templates.<sup>42</sup>
- **7.4. Dispersion Effect.** Dehydro-EGCG, the oxidation product of EGCG following the reduction step, could have an impact on the dispersion effect on the copper nanoparticles. The produced nanoparticles' stability and dispersion were preserved in part by this mechanism (Figure 10).<sup>42</sup>

#### 8. APPLICATIONS OF BTE-CAPPED NANOPARTICLES

**8.1. Biological Applications.** 8.1.1. MTT Method for Cell Viability. Cytotoxicity and cellular internalization of teacapped AuNPs revealed that the phytochemicals in tea provide the gold nanoparticles with a nontoxic coating. Even at high doses of T-AuNP-1, prostate (PC-3) and breast (MCF-7) cancer cells exhibited outstanding vitality, as evidenced by the MTT method for cell viability assessment. This suggests that the phytochemicals from tea coated on gold nanoparticles are safe and provide new possibilities for safe distribution and use in molecular imaging and treatment. 15

Green-synthesized AuNPs, derived from BTE extract, have a noteworthy potential effect on colon cancer cells. The outcome of the research showed that these nanoparticles, referred to as BTE-AuNPs, improved the chemotherapeutic medication doxorubicin's (DOX) ability to kill colon cancer cells. This improvement was made possible via a ROS-dependent mechanism that made the cancer cells more prone to apoptosis. When BTE-AuNPs and DOX were used together, colon cancer cells responded more violently to the combination than when DOX was used alone. The results of the study also suggested that the BTE-AuNPs could be strong doxorubicin chemosensitizers, which could have an impact on how well colon cancer treatment works. The results indicate that green-synthesized gold nanoparticles derived from BTE extract might increase doxorubicin's chemosensitivity in colon cancer patients.32

The cytotoxicity of the tea extracts synthesized AgNPs was assessed on Chicken embryo fibroblast (CEF) cells at concentrations of 9.37, 18.75, 37.5, 75, and 150  $\mu$ g/mL, respectively, using the MTT assay. The results presented in (Figure 11) demonstrated that the viability of the CEF cells was not substantially impacted by the presence of various [AgNPs] in comparison to the control group, which was not exposed to the nanoparticles. The difference in response between the untreated and treated cells was negligible, even at the greatest concentration of 150  $\mu$ g/mL of AgNPs.<sup>37</sup>

8.1.2. Antibacterial Activity. Methicillin-resistant Staphylococcus aureus (MRSA), Bacillus subtilis (B. subtilis), Escherichia coli (E. coli), and Staphylococcus aureus (S. aureus) were the microorganisms against which the antibacterial activity was evaluated. These microorganisms comprise antibiotic-resistant MRSA as well as Gram-positive and Gram-negative strains.

The biogenic silver nanoparticles had notable antibacterial action at a dosage of 100  $\mu$ g/mL: S. aureus: 90% fatality rate, MRSA: 75% fatality rate, B. subtilis: effects 75% deadly, *E. coli*: 80% fatality rate. Biogenic AgNPs are highly efficient against bacteria, including MRSA, which is resistant to antibiotics. They have potential uses as antibacterial agents on ceramic surfaces. BTE-AgNPs with a particle size of 26.9 nm exhibited antimicrobial action toward Gram + ve *Staphylococcus aureus*, while minimum antimicrobial effect was observed against Gram—ve *Escherichia coli*. The antibacterial action of the particles will be significantly influenced by their physicochemical properties, as demonstrated by Tang et al. <sup>58</sup>

The maximal antibacterial activity was demonstrated by nanoparticles with a plate-like morphology and a size of less than 10 nm. The occurrence of this phenomenon has been elucidated by Raza et al.,<sup>59</sup> who found that smaller nanoparticles exhibited greater antibacterial activity due to their increased stability and larger surface area. This resulted in

$$Cu^{2+} + 2e^{-} \longrightarrow Cu^{0} + Air(O_{2}) \longrightarrow CuO$$

Figure 10. A proposed mechanism for forming black tea-capped CuO nanoparticles. 42

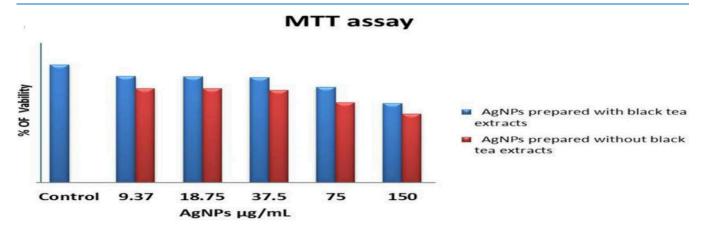


Figure 11. Viability of chicken embryo fibroblast cells after 72 h of incubation with AgNPs prepared with and without black tea extracts. Reprinted with permission from ref 37. Copyright 2017 World J. Exp. Biosci.

improved intracellular penetration of the bacterial cell walls and improved interactions. Additionally, in another study, the effect of particle size was demonstrated, as smaller particles exhibited greater activity against *Escherichia coli*. 60

AgNPs synthesized using black tea leaf extracts exhibited exceptional antibacterial properties against *Staphylococcus aureus* and Aspergillus species in contrast to FeNPs and CuNPs.<sup>38</sup>

AuNPs were discovered to possess antibacterial properties toward *Pseudomonas aeruginosa*, Vibrio cholera, *Staphylococcus aureus*, and *Escherichia coli*.<sup>34</sup> Consequently, black tea extract

can be employed as a natural reducing agent to generate AuNPs, AgNPs, FeNPs, and CuNPs which can be employed as an antibacterial medication to manage pathogenic bacteria.

8.1.3. Antifungal Activity of Metal Nanoparticles. The order of antifungal activity was as follows: Ag-NPs > Both Cu-NPs and Fe-NPs were synthesized using the extract of BTE leaves. Ag-NPs demonstrated a strong antifungal effect against fungal strains and the production of aflatoxins (AFs). Various authors reported that the spherical Ag-NPs exhibited robust antifungal activity in comparison to various metal NPs and commercially available antifungal agents. Similarly to the

antibacterial activity, the activity is likely to be well correlated with its decreased size and shape due to the increased surface area, which has an enhanced antifungal effect. Additionally, the antifungal activity of all metal nanoparticles synthesized in BTE leaf extract was not significantly different from one another at  $p \le 0.05$ .

Various authors have reported on the potential interaction mechanisms of NPs with fungal isolates. For example, Nasrollahi et al. <sup>61</sup> proposed that Ag-NPs may exert an effect on fungi by entering the cell membrane and influencing the respiratory chain. Consequently, the cessation of cell division led to cell mortality. Additionally, the inactivation of a protein and direct interaction with DNA were the results of NPs' interaction with fungi's protein. As a result, the interaction process results in mutation, which prevents DNA from replicating.

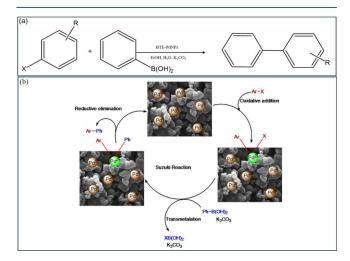
Furthermore, the nanoparticles often readily penetrate the cell wall due to their diminutive size. The cell lysis can be induced by this accretion in the cell membrane. Additionally, NPs can infiltrate the fungal spores by crossing the cell membrane. This results in forming an electron-light region in the cell's center, characterized by the presence of condensed DNA molecules and the detection of NPs within the cell. Subsequently, particulates disrupt the respiratory sequence, and ultimately, cell division is entirely obstructed, resulting in cell demise. 62,38

8.1.4. Anticancer Activity against He–La Cervical Cancer Cell Lines. The majority of cancer medications are indiscriminate in their destruction of both malignant and healthy cells, necessitating the development of innovative therapeutics. Nanotechnology is one of the most promising areas of inquiry in material science. The unique properties of AgNPs have the potential to significantly enhance the therapeutic efficacy of an anticancer agent, which is why they have considerable potential for development.<sup>22</sup>

Rajawat and Malik determined that the electrolytic deposition method could be employed to effectively synthesize highly pure silver nanoparticles with BTE extract as a capping agent. In comparison to silver nanoparticles that were capped with hazardous chemicals, those that were sealed with antioxidant polyphenols of black tea extract were discovered to be more environmentally benign and to have superior biomedical applications. The silver nanoparticles that were synthesized as-is exhibited a growth inhibition of nearly 70% for cervical cancer cell lines. The IC50 value was determined to be a 30-fold dilution of a 178 ppm concentration of the colloidal silver solution. <sup>22</sup>

- 8.1.5. Aflatoxins B1 Adsorption. The order of adsorption capability for all BT-NPs with AFB1 contamination was as follows: Fe-NPs > Cu-NPs > Ag-NPs. The equilibrium data indicated that the Langmuir isotherm was more favorable in terms of the adsorption capacity (131–139 ng/mg), Cu-NPs (114–118 ng/mg), and Ag-NPs (110–115 ng/mg). The adsorption process is endothermic, and spontaneous, and follows the pseudo-second-order, as indicated by kinetic studies and thermodynamic parameters. 38
- **8.2. Chemical Applications.** 8.2.1. Suzuki coupling Processes and the Reduction of 4-Nitrophenol. Suzuki-Miyaura is the name of the reaction between aryl halides and aryl boronic acids, which leads to the formation of a diverse array of unsymmetrical biaryls. <sup>63</sup> In an environmentally friendly medium, the BTE-PdNPs catalyst was employed as an efficient and heterogeneous catalyst for Suzuki coupling

reactions between phenylboronic acid and a variety of aryl halides (X = I, Br, Cl) and the reduction of 4-nitrophenol using sodium borohydride. The catalyst achieved excellent product yields with a diverse array of substrates and was recycled seven times without any significant loss of catalytic activity (Figure 12).<sup>47</sup>



**Figure 12.** (a, b) Suzuki coupling reaction in the presence of BTE-PdNPs. Reprinted with permission from ref 47. Copyright 2017 Elsevier.

**8.3. Environmental Applications.** 8.3.1. Degradation of Malachite Green (MG). Indeed, MG is a widely used cationic triphenylmethane dye that finds employment in a variety of sectors, including aquaculture, paints, inks, paper, leather, and textiles. However, because of its toxicity, MG might be removed from wastewater before being released into aquatic ecosystems due to serious environmental issues. 64–66

Using black tea iron nanoparticles derived from various tea extracts, it was discovered that the degradation efficiency of MG was 81.2%, 75.6%, and 67.1% for extracts of green tea, oolong tea, and BTE, respectively. These variations in the sizes and concentrations of FeNPs produced by the various tea extracts, as well as the FeNPs' reactivity, were responsible for the observed discrepancies in degradation efficiency. FeNPs made from green tea extracts had the best degradation efficiency, which was explained by the high content of caffeine and polyphenols, which served as capping and reducing agents throughout the FeNP synthesis process.

Several steps are involved in the FeNPs-based malachite green degrading mechanism that has been suggested. Malachite green is adsorbed onto iron oxide and nanoparticles in the initial step of the process. Reduction is the second procedure, where Fe<sup>0</sup> serves as the reducing agent. Malachite green's breakdown is also assumed to cleave the C = C- and = C=N-bonds that are connected to the benzene ring.<sup>44</sup>

8.3.2. Adsorption of Eriochrome Blue-Black B. BTE leaf extract was used as a green reducing agent to synthesize FeNPs at room temperature. The BTE leaf extract effectively reduced iron ions to FeNPs. The effectiveness of both unsupported FeNPs and FeNPs supported by bentonite (a type of clay) in removing Eriochrome Blue-Black B dye from wastewater. The effectiveness of dye removal was found to be directly influenced by the content of zero-valent iron. The study explored the impact of varying the zero-valent iron content from 0.5 to 2 g/L on dye removal efficiency. Increasing the

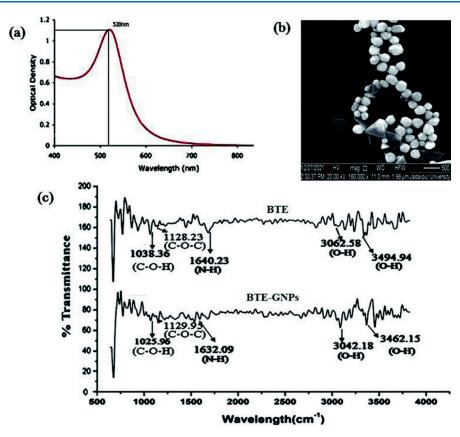


Figure 13. Characterization of BTE-AuNPs. (a) UV—visible absorption spectra for the reduced AuNPs of BTE. (b) SEM image of the BTE-AuNPs. (c) FT-IR spectra of both extract BTE and BTE extract-reduced AuNPs. Reprinted with permission from ref 32. Copyright 2022 The Royal Society of Chemistry.

zero-valent iron content led to an improved dye removal rate. This was attributed to an increase in surface sites available for reaction and a higher number of free electrons, which create more active sites. After 180 min, the decolorization efficiency improved significantly: Bentonite-supported FeNPs demonstrated a higher efficiency in removing the Eriochrome Blue-Black B dye from wastewater. 46

# 9. THE FATE OF BLACK TEA-CAPPED NANOPARTICLES

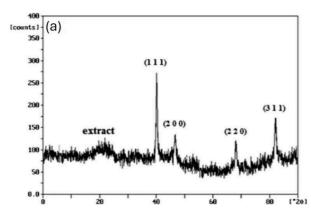
BTE leaf extract reduced an aqueous solution of HAuCl<sub>4</sub> to form the AuNPs. When the gold solution was decreased with a plant extract from BTE leaves, color changes in the reactive solution combination indicated the formation of AuNPs. HAucl<sub>4</sub> in an aqueous solution maintained its yellow hue, indicating that Au<sup>3+</sup> was not lowered. It became pale pink once 1 mL of black tea extract was added, instead of light yellow. This demonstrated the Au<sup>3+</sup> was reduced to Au<sup>0</sup>. This finding demonstrated using BTE leaf extract to reduce and cap, or stabilize, freshly generated metal AuNPs. It is known that the maximum absorbance of gold nanoparticles is between max 400 and 700 nm.<sup>32</sup>

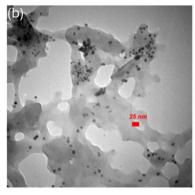
The BTE extract's FTIR spectrum exhibited peaks at 3494.94 cm<sup>-1</sup> (O–H stretching of carboxylic acid, strong peak intensity), 3062.58 cm<sup>-1</sup> (O–H stretching of carboxylic acid, medium peak intensity), 1640.23 cm<sup>-1</sup> (N–H bend of amines, strong peak intensity), 1128.23 cm<sup>-1</sup> (C–O–C stretching of polysaccharides, strong peak intensity), and 1038.36 cm<sup>-1</sup> (C–O–H stretching of amino acids, strong peak intensity). The gold nanoparticles' FTIR spectrum revealed

peaks at 3462.15 cm<sup>-1</sup> (O–H stretching of carboxylic acid, strong peak intensity), 3042.15 cm<sup>-1</sup> (O–H stretching of carboxylic acids, strong peak intensity), 1632.09 cm<sup>-1</sup> (N–H stretching of amines, medium peak intensity), 1129.95 cm<sup>-1</sup> (C–O–C stretching of polysaccharides, strong peak intensity), and 1025.96 cm<sup>-1</sup> (C–O–H stretching of amino acids, strong peak intensity).<sup>32</sup>

Das et al. proposed that the reduction of HAuCl<sub>4</sub> to reduced gold nanoparticles may be attributed to functional groups such as O–H, N–H, C–O–C, and COOH. The peaks of black tea AuNPs exhibit a modest displacement from the peaks of black tea extract, as evidenced by the comparison of 2942.18 cm<sup>-1</sup>, 2942.18 cm<sup>-1</sup>, and 2859.64 cm<sup>-1</sup>. The same functional groups are present in both black tea extract and black tea-capped AuNPs (Figure 13).<sup>32</sup>

XRD spectroscopy was employed to ascertain the crystalline character of the green synthesized BTE-PdNPs catalyst. The face-centered cubic (fcc) crystalline structure was indexed for metallic palladium (Pd $^0$ ) according to crystallographic planes, as evidenced by the four distinct reflections at 39.7 $^\circ$  (1 1 1), 46 $^\circ$  (2 0 0), 68 $^\circ$  (2 2 0), 82 $^\circ$  (311), and 86 $^\circ$  (2 2 2). TEM was employed to investigate the particles' crystallinity, size, and morphology, synthesized from BTE leaf extract. The majority of the particles were nearly spherical, with a size range of 5 to 8 nm. The accumulation of smaller nanoparticles resulted in the formation of slightly larger, nonspherical particles. The biopolymer layer that encircled the dispersed colloidal PdNPs was intriguing, as it appeared to be responsible for the nanoparticles' reduction and stabilization (Figure 14).





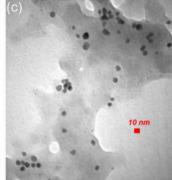


Figure 14. Characterization of BTE-PdNPs. (a) XRD pattern of BTE-PdNPs. (b, c) represents TEM images of the BTE-PdNPs. Reprinted with permission from ref 47. Copyright 2017 Elsevier.

# 10. EFFECT OF COMPONENTS

To understand the critical roles of the various catechins present in BTE leaves on the overall reduction of NaAuCl $_4$  to the corresponding AuNPs, Nune et al. 15 performed a series of independent experiments using directly the commercially available family of catechins which include: catechin, epicatechin, epicatechin gallate, catechin gallate, epigallocatechin, and epigallocatechin gallate.

Podstawczyk et al.<sup>67</sup> demonstrated a route of size-controllable synthesis of CuNPs using (+)-catechin solution and the chemistry behind the reaction. Lu et al.<sup>68</sup> conjugated epigallocatechin-3-gallate (EGCG), a significant component of tea catechins, with nanoparticles and evaluated as an anticancer agent. The green synthesis of AuNPs was described as eco-friendly, utilizing catechin as a reducing and capping agent. The reaction was completed within 1 h at room temperature without any external energy, and an exceptional yield (99%) was achieved, as confirmed by inductively coupled plasma mass spectrometry. These gold nanoparticles were synthesized in various configurations, with an estimated diameter of 16.6 nm. <sup>69</sup> Epicatechin-capped AgNPs were investigated as a sensor for Pb<sup>2+</sup> in water and blood samples.<sup>70</sup> Epigallocatechin gallate-loaded Chitosan nanoparticles were synthesized and characterized by Ponnuraj et al. through the ionic-gelation method. Chavva compared the antitumor efficacies of AuNPs synthesized using the conventional method of citrate-AuNPs and using EGCG as a reducing agent (E-GNPs) in a diversity of established cancer cell lines. A highperformance nanocomposite sensor has been created based on screen-printed electrodes (SPE). This sensor comprises tungsten disulfide (WS2) grains decorated with catechincapped AuNPs and carbon black (CB). The simultaneous determination of hydroxycinnamic acid (hCN) structural analogs: caffeic (CF), sinapic (SP), and p-coumaric acids (CM) is facilitated by the exceptional antifouling features of WS2 with gold nanoparticles-catechin into a higher conductivity network of CB, contributing exclusive selectivity as well as sensitivity.

#### 11. CONCLUSIONS AND FUTURE PERSPECTIVE

Synthesis of metal and metal oxide nanoparticles requires BTE as a reducing and/or capping agent. Moreover, BTE-metal nanoparticles of hydrosol are used for anticancer, antifungal, cytotoxicity, cell viability, and Aflatoxins B1 adsorption without the purification of BTE capping agent. We can consider it a

direct application of BTE in nanotechnology. BTE-capped nanoparticles are also washed and dried for the application of dye removal. It can be regarded as an indirect application. BTE is a natural phytochemical, consisting of Polyphenols are the most prevalent compounds, with catechins being the most prevalent. Epigallocatechin gallate (EGCG), epigallocatechin (EGC), epicatechin gallate (ECG), and epicatechin (EC) are the most significant catechins Among them, catechins and EGCG are reported to be passivate metal nanoparticles independently. Thus, catechins and EGCG can be considered responsible polyphenols for forming metal nanoparticles. Moreover, BTE is cheap, easily available, and nontoxic. So, the involvement of BTE often makes the process green and environmentally friendly. Such advantageous features make BTE an important candidate for medicinal technology and sustainable water management. Our review article may be an asset for young scientists, venturing into the field of green nanotechnology and environmental science. This review article fills the gap between nanometric and non-nanometric applications of a kitchen chemical.

# ASSOCIATED CONTENT

# **Data Availability Statement**

No data are associated with the manuscript.

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