

# Quantitative Analysis of Titanium Dioxide (TiO<sub>2</sub>) Levels as Food Additives in Various Food Samples from the Jordanian Market

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*This study presents a quantitative determination of titanium dioxide (TiO<sub>2</sub>, E171) in food products frequently consumed by children. An optimized acid digestion method using concentrated sulfuric acid, nitric acid, and hydrogen peroxide at 200 °C for 45 minutes was developed to efficiently extract TiO<sub>2</sub> from food matrices. The recovered TiO<sub>2</sub> was quantified using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and UV-Visible spectrophotometry. Both methods were successfully applied to twenty-five food products from the Jordanian market, including chewing gums, candies, and powdered juices. Structural and morphological characterization of the extracted TiO<sub>2</sub> was carried out using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Dynamic Light Scattering (DLS). XRD confirmed the presence of both anatase and rutile crystalline phases, while SEM revealed irregular particle morphology. FTIR spectra showed characteristic Ti–O vibrations, and DLS analysis indicated that the particle sizes were in the micrometer range, with no evidence of engineered nanoparticles. The proposed approach enables accurate monitoring of TiO<sub>2</sub> content in foods and supports regulatory efforts to evaluate consumer exposure to E171, particularly in products marketed to children.*

**Keywords:** titanium dioxide; E171; food additives; ICP-OES; UV-Visible spectrophotometry

## Introduction

Titanium dioxide (TiO<sub>2</sub>) is an oxide of titanium, commonly found in nature in the form of the mineral polymorphs brookite, rutile, and anatase. Due to its exceptionally high refractive index and strong light-scattering properties, TiO<sub>2</sub> is widely used as a white pigment, known commercially as “titanium white” [1]. These optical characteristics have led to its extensive incorporation in sunscreens and cosmetic formulations, where it acts as a physical UV filter by reflecting and scattering incident light.

In the food industry, food-grade TiO<sub>2</sub> (designated as E171) has been used for decades as a whitening and brightening agent in a variety of products such as confectioneries, chewing gums, candies, and chocolates [1,2]. It is also commonly present in coatings, pharmaceuticals, toothpaste, and other personal care products [3]. Recent industrial developments have favored the use of TiO<sub>2</sub> in its nanosized form, owing to enhanced functional properties. Global TiO<sub>2</sub> production is estimated at approximately 5.1 million tonnes per year, with expectations for continued growth [4,5].

However, growing concerns regarding the potential health risks associated with nano-TiO<sub>2</sub> exposure have prompted increased scrutiny. Toxicological studies have reported adverse biological effects of E171 nanoparticles, including immune responses, tissue necrosis, renal apoptosis, and inflammation [6,7]. The International Agency for Research on Cancer

(IARC) has classified TiO<sub>2</sub> as “possibly carcinogenic to humans.” In response to these findings, France implemented a national ban on the use of E171 in food products beginning in 2020 [8–11].

Various analytical techniques have been employed for the detection and quantification of TiO<sub>2</sub> in food matrices. Among the most widely used methods is inductively coupled plasma optical emission spectrometry (ICP-OES), which enables multi-elemental analysis with high precision [12]. For instance, Sharif et al. [13] analyzed TiO<sub>2</sub> concentrations in a variety of conventional Middle Eastern food products, including tahini, halvah, canned chickpeas, jams, chewing gums, and juice powders. While TiO<sub>2</sub> was absent in some of these items, in agreement with product labeling, its presence in chewing gum and juice powders was confirmed at concentrations below the regulatory threshold of 1%, using UV-Visible spectrophotometry.

In many previous studies, digestion procedures based on hydrofluoric acid (HF) and nitric acid (HNO<sub>3</sub>) have been applied to break down the crystalline lattice of TiO<sub>2</sub> prior to instrumental analysis [3,14]. However, the use of HF poses significant safety and equipment compatibility challenges. In this study, we propose an alternative digestion protocol utilizing a safer and more accessible reagent system composed of concentrated sulfuric acid (98%), nitric acid (65%), and hydrogen peroxide (30%) under controlled thermal conditions (200 °C). This modified digestion approach facilitates

the efficient breakdown of TiO<sub>2</sub> in food matrices without the need for specialized HF-resistant equipment.

Elsewhere ICP-OES and UV-Vis spectrophotometry, other advanced techniques such as inductively coupled plasma mass spectrometry (ICP-MS) [15] and Raman spectroscopy [16] have also demonstrated high sensitivity and selectivity for TiO<sub>2</sub> detection, especially at trace levels in complex matrices. Raman spectroscopy, in particular, benefits from the Raman-active characteristics of titanium dioxide crystal structures.

The present study aimed to evaluate the TiO<sub>2</sub> content in a selection of widely consumed food products in the Jordanian market using two independent and validated analytical techniques - ICP-OES and UV-Vis spectrophotometry. Additional objectives included the optimization of sample digestion protocols, comparison of method performance, and physicochemical characterization of the extracted TiO<sub>2</sub> using complementary techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and dynamic light scattering (Zetasizer).

## Experimental part

### *Chemicals and Reagents*

The following analytical-grade chemicals and reagents were used throughout the study without further purification. Ammonium sulfate (98.5% purity) was obtained from CHEMLAB (West Yorkshire, England). Concentrated sulfuric acid (98%) was supplied by Industrial Estate, Worli Road. Nitric acid (65% w/w) was sourced from Guangdong Guanghua Chemistry Factory Co., China, and hydrogen peroxide (30% w/w) was purchased from Merck (Germany). Double-distilled water was prepared in-house using a GFL single-distillation unit. A certified titanium standard solution (1000 ppm, Ti(IV), 99.9% purity) was obtained from Acros Organics (Geel, Belgium).

### *Instruments*

Spectral measurements were conducted using a UV-300 double-beam UV-Vis spectrophotometer (Shimadzu, Japan) across 190–1100 nm with 1 cm matched quartz cuvettes. Ti(IV) concentrations were quantified by ICP-OES (Thermo Scientific, USA). SEM imaging was performed using a Phenom XL G2 SEM (Thermo Fisher Scientific, USA) and a high-resolution Apreo 2S LoVac field emission SEM with an EDS Ultra Dry detector and Pathfinder software. Particle size was assessed using a Zetasizer Nano ZS (Malvern Instruments, UK). XRD was carried out using a D8 Advance diffractometer (Bruker, Germany) with Ni-filtered Cu K $\alpha$  radiation. FT-IR spectra were acquired on an FTIR-7600 (Lambda Scientific, Australia) in the range 4000–400 cm<sup>-1</sup>.

### *UV-Vis Spectrophotometry*

#### *Ti(IV) standard preparation*

A stock Ti(IV) solution (300 mg L<sup>-1</sup>) was prepared by dissolving an appropriate amount of titanium in a mixture of 10 mL sulfuric acid and 4 g ammonium

sulfate, followed by heating to produce white fumes. After cooling, the solution was diluted to 100 mL with double-distilled water. A 150 mM hydrogen peroxide solution was prepared from a 30% H<sub>2</sub>O<sub>2</sub> stock. An inorganic solvent mixture was made by dissolving 4 g ammonium sulfate and 10 mL sulfuric acid in 100 mL deionized water. Under optimized conditions, 1.5 mL of H<sub>2</sub>O<sub>2</sub> was mixed with Ti(IV) aliquots in 10 mL flasks, diluted with the inorganic solvent, and measured at 407 nm.

### *Food sample preparation*

All selected food samples were purchased from local Jordanian markets and categorized into four groups: snacks, chocolates, chewing gum, and candies. Priority was given to products explicitly listing TiO<sub>2</sub> on their labels; however, samples exhibiting characteristic white coloration, despite lacking label indications, were also included. The full list of investigated samples is presented in Table S1.

To eliminate organic content, the samples were subjected to incineration. Measured portions of each sample were placed in porcelain crucibles and ashed in a muffle furnace at 600 °C for 2 hours. After cooling to room temperature, acid digestion was performed on the carbon-free ash. Specifically, 10 mL of nitric acid (65%) and 5 mL of concentrated sulfuric acid were added to the residue in a beaker and heated in a water bath until dissolution commenced. The solution was then transferred to a hotplate and heated at 200 °C until dense nitric acid fumes were released, ensuring complete digestion. Subsequently, an additional 5 mL of sulfuric acid and 4 g of ammonium sulfate were added, resulting in the evolution of white vapors indicative of TiO<sub>2</sub> solubilization. The digest was cooled, quantitatively transferred to a 100 mL volumetric flask, and diluted to volume with double-distilled water for analysis.

### *ICP-OES Measurements*

#### *Ti (IV) standard preparation*

The pure titanium standard solution was prepared by diluting an appropriate amount in concentrated sulfuric acid, which was then diluted with distilled water to yield a 500-ppm final concentration. Following the preparation of the above-mentioned standard solution, further dilution resulted in the following concentrations: 0.1, 0.3, 0.7, 1, 2, 5, 10, and 20 ppm [3]. The preparation and dilution were carried out carefully to present accurate results and maintain homogeneity for all concentrations. The operational conditions are represented in Table S2.

### *Food sample preparation*

A sample of 1 to 20 g  $\pm$  0.1 g was taken in a porcelain dish. Then, the sample was placed in the furnace at a temperature of 600°C and set to ash for a minimum period of 4 hours. The sample was then allowed to cool, and about 3 g of ammonium sulfate was added to the ash and transferred to a conical flask. Then, 20 mL of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) was added, and the solution was heated on a hotplate. It was kept to boil

until it became clear. Afterward, the solution was left to cool down to room temperature, after which 10 mL of distilled water was added slowly. The mixture was then transferred quantitatively to a 100 mL volumetric flask and diluted to the mark with deionized water [18].

#### *SEM Measurement*

The extracts from the selected samples were dried in ovens at low temperatures (60–80°C), sufficient to eliminate moisture without interfering with the structure of the food. The dried samples were then finely ground to a homogeneous powder.

#### *Zetasizer nano measurement*

The extract from the selected samples was then ground to obtain a fine, homogeneous powder. Next, 0.1 grams of the sample was weighed and dissolved in 10 mL of distilled water (H<sub>2</sub>O) to prepare a suspension.

#### *FTIR measurement*

The extract from the selected samples started with the low-temperature drying of the sample at 60–80 °C, followed by grinding of the sample into a fine powder to obtain a homogenous powder. For the preparation of the disks, a sample of about 1–2 mg was mixed with pure KBr (about 100–200 mg), an infrared transparent material, and was pressed using a hydraulic press into a transparent disk.

#### *XRD measurement*

The extract from the selected samples was first fired in the furnace at 600°C. It is then ground into a fine, homogeneous powder, which improves particle homogeneity and yields a better diffraction pattern.

#### *Statistical analysis*

All measurements were conducted in triplicate and reported as mean ± standard deviation. Relative standard deviations were below 2%, considered acceptable. One-way ANOVA was applied to evaluate differences among sample groups ( $p < 0.05$ ) using Microsoft Excel 2017.

## Results and Discussion

### **ICP-OES analysis**

**Linearity and Range.** Calibration for Ti(IV) was performed using eight standard solutions (0.1–20.0 mg L<sup>-1</sup>). The resulting calibration curve exhibited excellent linearity with a correlation coefficient ( $R^2$ ) of 0.9997 (Figure S1).

**Limit of Detection and Quantification.** The emission line at 336.1 nm was selected due to its high sensitivity and minimal spectral interference. The LOD and LOQ were calculated using the standard deviation of blank measurements and the slope of the calibration curve, yielding values of 0.51 mg L<sup>-1</sup> and 1.56 mg L<sup>-1</sup>, respectively.

**Precision.** Repeatability was assessed through triplicate analysis of samples, with relative standard deviation (RSD) values ranging from 0.14% to 0.50%, indicating high precision across the tested concentration range.

### **UV-Visible spectrophotometry**

**Linearity and Range.** Ti(IV) detection by UV-Vis

spectroscopy at 407 nm was calibrated using thirteen standard solutions (5–100 mg L<sup>-1</sup>). The resulting calibration curve yielded  $R^2 = 0.9994$  (Figure S2).

**Limit of Detection and Quantification.** The LOD and LOQ were determined to be 1.00 mg L<sup>-1</sup> and 3.04 mg L<sup>-1</sup>, respectively, demonstrating acceptable sensitivity.

**Precision.** RSD values ranged from 0.30% to 1.80%, confirming adequate analytical precision for Ti(IV) quantification in food matrices.

### **Quantification of TiO<sub>2</sub> in commercial food products**

Following method validation, twenty-five food products labeled as containing the whitening agent E171 were analyzed using both ICP-OES and UV-Vis spectrophotometry. The TiO<sub>2</sub> concentrations ranged from 10 to 40 mg L<sup>-1</sup>, corresponding to 0.001% to 0.004% (w/w), which are well below the maximum permissible limit of 1% established by the U.S. Food and Drug Administration for food-grade titanium dioxide. The detailed results are summarized in Table 1.

No statistically significant differences were observed between the TiO<sub>2</sub> concentrations measured by ICP-OES and those obtained by UV-Vis analysis. Likewise, the calibration slopes and analytical recoveries for the two methods were comparable. This contrasts with previous studies [1], where ICP-OES tended to underestimate TiO<sub>2</sub> content, possibly due to sample matrix effects or instrumental calibration inconsistencies - issues that were not observed in the present analysis.

Each sample was analyzed in triplicate, and results are reported as mean ± standard deviation. The RSD values for both analytical methods were below 2%, indicating satisfactory precision. Statistical evaluation using one-way ANOVA (performed in Microsoft Excel 2017) confirmed the absence of significant intra-group differences ( $p > 0.05$ ), further validating the consistency and reproducibility of the results.

In comparing the two analytical techniques, ICP-OES demonstrated superior sensitivity, lower detection limits, and the added advantage of multi-elemental detection, which reduces overall analysis time and sample preparation workload. However, UV-Vis spectrophotometry remains advantageous for routine screening due to its operational simplicity and lower cost. Moreover, it requires consistent sample mass (1 g) and is readily adaptable to quality control settings in food laboratories.

Together, the results confirm the suitability of both ICP-OES and UV-Vis spectrophotometry for accurate determination of TiO<sub>2</sub> in food products, with the choice of method depending on the specific requirements of sensitivity, throughput, and available resources.

### **Morphological and structural characterization of TiO<sub>2</sub> in the food samples**

#### **SEM-EDX Analysis**

The surface morphology and elemental

**Table 1.** Comparison of titanium dioxide (TiO<sub>2</sub>) content in selected food products determined by ICP-OES and UV–Visible spectrophotometry.

| Sample No. | Samples                | TiO <sub>2</sub> (mg L <sup>-1</sup> ) ± SD |            | TiO <sub>2</sub> % (W/W) |            |
|------------|------------------------|---------------------------------------------|------------|--------------------------|------------|
|            |                        | ICP-OES                                     | UV-Visible | ICP-OES                  | UV-Visible |
| 1          | Chewing gum 1          | 33.7 ± 0.3                                  | 31.3 ± 0.5 | 0.003                    | 0.003      |
| 2          | Chewing gum 2          | 38.9 ± 0.4                                  | 32.6 ± 0.5 | 0.004                    | 0.003      |
| 3          | Chewing gum 3          | 35.8 ± 0.3                                  | 33.8 ± 0.5 | 0.004                    | 0.003      |
| 4          | Tahina                 | 17.8 ± 0.2                                  | 16.6 ± 0.9 | 0.002                    | 0.002      |
| 5          | Powder juice (Shmpart) | 15.1 ± 0.2                                  | 9.4 ± 1.0  | 0.002                    | 0.001      |
| 6          | Powder juice (Squeez)  | 13.0 ± 0.2                                  | 13.3 ± 0.9 | 0.001                    | 0.001      |
| 7          | Powder juice (Tang)    | 12.8 ± 0.5                                  | 9.0 ± 1.2  | 0.001                    | 0.001      |
| 8          | Milk Cake              | 21.8 ± 0.5                                  | 23.8 ± 0.5 | 0.002                    | 0.002      |
| 9          | Coffee Creamer         | 24.7 ± 0.9                                  | 24.5 ± 0.5 | 0.002                    | 0.002      |
| 10         | Powder drink           | 13.2 ± 0.4                                  | 9.2 ± 1.0  | 0.001                    | 0.001      |
| 11         | Fruits Skittles        | 16.7 ± 0.4                                  | 15.8 ± 0.9 | 0.002                    | 0.002      |
| 12         | Halawa                 | 27.9 ± 0.6                                  | 25.0 ± 0.5 | 0.003                    | 0.003      |
| 13         | Mentos                 | 9.0 ± 0.4                                   | 11.1 ± 1.0 | 0.001                    | 0.001      |
| 14         | Hummus                 | 11.5 ± 0.3                                  | 12.0 ± 1.0 | 0.001                    | 0.001      |
| 15         | Marshmallows           | 8.5 ± 0.7                                   | 10.6 ± 1.0 | 0.001                    | 0.001      |
| 16         | White chickpeas        | 19.3 ± 0.4                                  | 15.8 ± 0.9 | 0.002                    | 0.002      |
| 17         | Gravel chocolate       | 20.8 ± 0.3                                  | 24.4 ± 0.5 | 0.002                    | 0.002      |
| 18         | Jelly candy            | 18.8 ± 0.4                                  | 16.4 ± 0.9 | 0.002                    | 0.002      |
| 19         | Cake ornament          | 24.0 ± 0.9                                  | 25.6 ± 0.5 | 0.003                    | 0.003      |
| 20         | Candy                  | 14.3 ± 0.6                                  | 12.8 ± 0.5 | 0.001                    | 0.001      |
| 21         | Nescafe 2in 1          | 15.6 ± 0.7                                  | 13.2 ± 0.9 | 0.001                    | 0.001      |
| 22         | Powder juice (Darina)  | 14.8 ± 0.6                                  | 9.2 ± 1.0  | 0.001                    | 0.001      |
| 23         | Salad dressing         | 9.3 ± 0.8                                   | 9.9 ± 1.0  | 0.001                    | 0.001      |
| 24         | Wafer                  | 6.9 ± 0.6                                   | 8.0 ± 1.0  | 0.001                    | 0.001      |
| 25         | Bread cakes            | 11.5 ± 0.8                                  | 10.9 ± 1.0 | 0.001                    | 0.001      |

composition of TiO<sub>2</sub> particles present in selected food samples were investigated using Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDX). As shown in Figure 1, the titanium dioxide particles extracted from a chewing gum sample displayed irregular shapes, heterogeneous sizes, and uneven surface distribution. This morphological pattern is typical of food-grade TiO<sub>2</sub> (E171), which is not monodispersed and may originate from natural mineral sources or be intentionally blended from different crystal forms to optimize whiteness and opacity [1, 3].

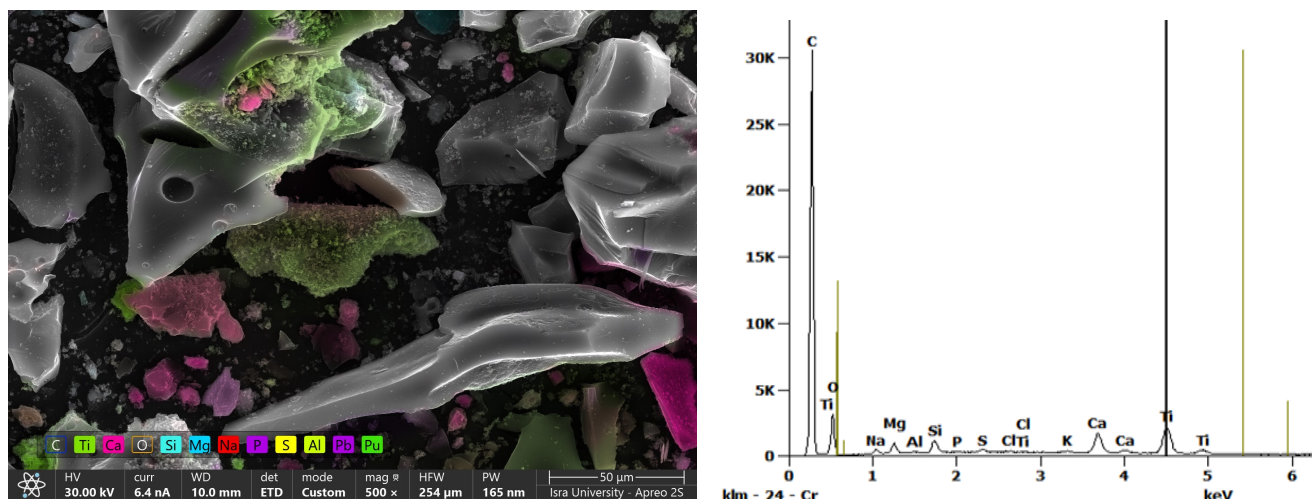
EDX analysis confirmed the presence of titanium as a major element, along with other minor constituents possibly originating from the food matrix. The elemental fingerprint matched the expected profile for TiO<sub>2</sub>, in agreement with previous studies that employed SEM–EDX to confirm the presence of engineered nanomaterials in complex food matrices [1, 6, 12]. Such morphology also suggests that TiO<sub>2</sub> was not uniformly distributed throughout the product,

indicating that its incorporation into food formulations may not be tightly controlled.

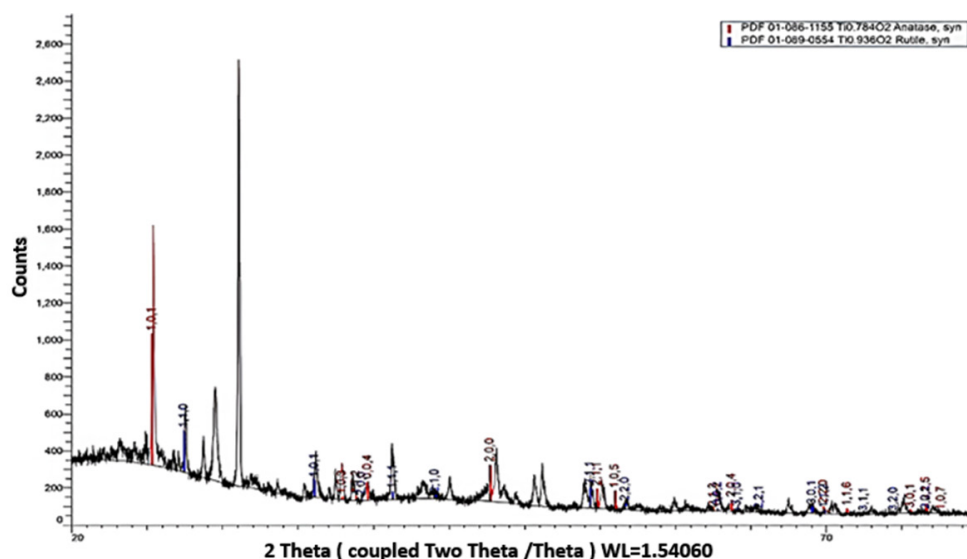
#### X-ray Diffraction (XRD) Analysis

X-ray diffraction analysis was performed to determine the crystal structure of TiO<sub>2</sub> particles in the chewing gum sample, selected based on its high Ti content. As shown in Figure 2, the diffraction pattern revealed distinct peaks corresponding to both the anatase and rutile crystalline phases of TiO<sub>2</sub>, which are polymorphic forms sharing a tetragonal system but differing in thermodynamic stability and optical properties [15].

Anatase is known to form at lower temperatures and tends to exhibit higher photocatalytic activity, while rutile is the more thermodynamically stable phase at elevated temperatures. The simultaneous presence of both phases suggests that the TiO<sub>2</sub> used in the food additive may have been subjected to partial thermal transformation or that a mixed-phase commercial pigment was utilized intentionally. This phenomenon has been reported in prior studies that examined



**Figure 1.** Scanning Electron Microscopy (SEM) image and corresponding Energy Dispersive X-ray (EDX) spectrum of a chewing gum sample.



**Figure 2.** X-ray diffraction (XRD) pattern of a chewing gum sample, revealing the crystalline structure and confirming the presence of anatase and rutile phases of titanium dioxide ( $\text{TiO}_2$ ).

industrial-grade  $\text{TiO}_2$  used in consumer products [6, 12]. The detection of both crystalline forms reinforces the need for crystallographic characterization in addition to elemental analysis when assessing additive compliance with regulatory requirements.

#### Dynamic Light Scattering (DLS) Analysis

To assess the hydrodynamic size distribution of  $\text{TiO}_2$  particles, samples of food-grade  $\text{TiO}_2$  and selected food extracts (e.g., Mentos and wafer) were analyzed using Dynamic Light Scattering (DLS). As illustrated in Figure S3, pure  $\text{TiO}_2$  displayed a high mean particle diameter of approximately 8155 nm with an extremely narrow distribution, indicating good uniformity and potential aggregation into larger structures. By contrast, the Mentos extract (Figure S4) exhibited a mean hydrodynamic diameter of 1667 nm, accompanied by a standard deviation of 163.3 nm, reflecting significant polydispersity and variability in particle dispersion.

These findings align with previously reported results showing that food-grade  $\text{TiO}_2$  particles often fall within the micrometer scale and tend to form aggregates in aqueous suspensions [3, 6]. According to the European Commission's definition, particles must be under 100 nm to be considered "nanomaterials." Hence, the tested samples would not meet the criteria for engineered nanomaterials, which is relevant for toxicological classification and regulatory status [5, 16].

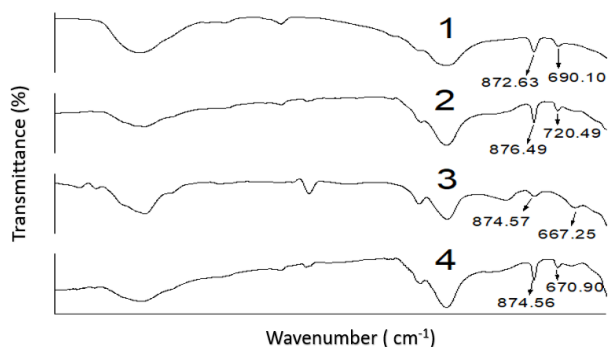
DLS results also highlight the influence of the food matrix on particle stability. In sugary or lipid-rich environments,  $\text{TiO}_2$  particles may aggregate or be partially embedded in the matrix, making their individual size and shape less accessible to direct measurement. This factor underlines the importance of combining multiple characterization techniques to overcome matrix-related analytical challenges [12].

### Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectroscopy was employed to qualitatively identify the presence of TiO<sub>2</sub> in food samples based on their vibrational signatures. The FTIR spectra of pure TiO<sub>2</sub> and three selected food samples (jelly candy, Darina juice, and coffee creamer) are shown in Figure 3. All spectra exhibited absorption bands in the range of 400–900 cm<sup>-1</sup>, which correspond to Ti–O and Ti–O–Ti stretching vibrations. Specifically, a weak but characteristic band at ~690 cm<sup>-1</sup> was attributed to the anatase form, while a more pronounced band near 873 cm<sup>-1</sup> is indicative of rutile [19].

Additional spectral features included broad bands at 3200–3550 cm<sup>-1</sup>, corresponding to hydroxyl (O–H) group vibrations, and peaks at 1600–1630 cm<sup>-1</sup>, associated with water bending modes. These signals suggest the presence of water molecules or hydrogen-bonded organic species interacting with the surface of TiO<sub>2</sub> particles. Shifts in peak intensity and position among food samples reflect potential interactions of TiO<sub>2</sub> with matrix components such as carbohydrates, fats, and proteins. Similar FTIR signatures for food-incorporated TiO<sub>2</sub> have been described in previous work [6, 19].

Importantly, FTIR was not only effective in identifying TiO<sub>2</sub> qualitatively but also helped reveal differences in matrix composition that may affect particle dispersion, chemical interactions, and ultimately bioavailability. The absence of significant interfering peaks in the fingerprint region supports the feasibility of FTIR as a rapid screening tool in food quality control settings.



**Figure 3.** Fourier Transform Infrared (FTIR) spectra of four samples: (1) pure titanium dioxide (TiO<sub>2</sub>), (2) jelly candy, (3) Darina juice, and (4) coffee creamer.

The morphological and structural characteristics of titanium dioxide in commercially available food products were comprehensively evaluated using complementary techniques including SEM–EDX, XRD, DLS, and FTIR. The results confirmed that the TiO<sub>2</sub> found in these samples: exists primarily in the micrometer size range; contains both anatase and rutile crystalline phases; is heterogeneously distributed within the food matrix; exhibits spectral

features consistent with interaction with organic food components.

These findings are consistent with prior studies reporting on the form, structure, and behavior of TiO<sub>2</sub> in food products. Moreover, the observed variability in particle morphology and size underlines the need for standardized methods of TiO<sub>2</sub> characterization to ensure compliance with labeling and regulatory limits, especially in regions where E171 use is restricted or banned. The multi-method approach employed here demonstrates the value of combining morphological, crystallographic, spectroscopic, and particle size analyses to obtain a complete profile of food additives such as titanium dioxide.

In Jordan, the regulation of food additives, including titanium dioxide (E171), falls under the jurisdiction of the Jordan Standards and Metrology Organization (JSMO) and the Jordan Food and Drug Administration (JFDA), which are responsible for ensuring food safety and consumer protection. In 2022, based on safety concerns raised by the European Food Safety Authority (EFSA), the JFDA officially prohibited the use of E171 in food products and updated its national list of approved additives to align with international standards. Jordanian Standard No. 2232 mandates the full disclosure of all ingredients on product labels, thereby enhancing transparency and enabling informed consumer choices. Enforcement mechanisms include routine inspections of food items available on the market and collaboration with the Jordanian Customs Department to prevent the importation of non-compliant products.

Despite these regulatory efforts, the present study confirmed the continued presence of E171 in several food products available in the Jordanian market. This discrepancy between regulation and market reality underscores significant challenges in enforcement. Factors contributing to non-compliance include insufficient monitoring capacity, limited enforcement resources, delays in updating national systems, and a general lack of awareness among both consumers and manufacturers. Moreover, the heavy reliance on imported processed foods, which may not be reformulated to meet Jordanian standards, further complicates regulatory enforcement.

Notably, regulatory clarity remains an issue. While the 2022 ban officially restricts the use of E171 in foods, representatives of the JFDA have stated that titanium dioxide is not universally banned—neither globally nor within Jordan—and continues to be permitted for use in certain non-food applications, such as cosmetics and pharmaceutical formulations, where it functions primarily as a pigment.

By contrast, the Saudi Food and Drug Authority (SFDA) has taken a more stringent approach. In August 2024, it issued a circular banning the use of titanium dioxide in all food products. This directive includes prohibitions on imports, production inputs, and the renewal of previously granted approvals. The SFDA

emphasized that the decision was precautionary, given the lack of definitive evidence categorizing TiO<sub>2</sub> as carcinogenic or directly harmful to human health.

The observed persistence of E171 in the Jordanian market despite its official ban reflects systemic limitations in regulatory implementation and enforcement. These findings highlight the need for enhanced monitoring infrastructure, greater regulatory coordination at national and international levels, and improved public communication to ensure alignment between legislative intent and market practices.

## Conclusion

This study evaluated the presence and concentration of titanium dioxide (TiO<sub>2</sub>, E171) in 25 commonly consumed food products available on the Jordanian market. Despite the regulatory ban on E171 imposed by the Jordan Food and Drug Administration (JFDA) in 2022, TiO<sub>2</sub> was detected in all analyzed samples. Importantly, the measured concentrations were within the internationally accepted safety threshold of less than 1% (w/w), as established by the U.S. Food and Drug Administration (FDA). However, only six products explicitly listed TiO<sub>2</sub> on their ingredient labels, raising concerns regarding labeling transparency and regulatory compliance.

Several factors may account for the continued presence of TiO<sub>2</sub> in food items, including legacy stock produced prior to the implementation of the ban, the use of imported ingredients or pre-manufactured components containing TiO<sub>2</sub>, cross-contamination during manufacturing, or incomplete enforcement of the regulatory directive.

For the first time in the context of the Jordanian market, this study employed Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) in parallel with UV-Visible spectrophotometry to quantify TiO<sub>2</sub>. Both techniques demonstrated strong analytical performance and consistency, validating their suitability for detecting low levels of titanium dioxide in complex food matrices. The methods yielded precise and reproducible results, with all values confirmed to be below the established safety limits.

Qualitative assessments using Scanning Electron Microscopy (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) revealed that TiO<sub>2</sub> particles in the food samples were irregularly shaped and unevenly distributed, while X-ray Diffraction (XRD) analysis of a chewing gum sample identified both anatase and rutile crystalline phases of TiO<sub>2</sub>. Particle size distribution, assessed via Dynamic Light Scattering (DLS), indicated that the TiO<sub>2</sub> materials were in the micrometer rather than nanometer range.

This study highlights the importance of implementing robust and sensitive analytical methods for monitoring regulated additives such as E171. It also underscores the need for improved regulatory enforcement, comprehensive product screening, and clear labeling requirements to ensure consumer safety

and industry accountability in Jordan and similar regulatory environments.

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## Disclosure statement

The authors declare no competing interests.

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