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Water activity and physicochemical variability in dietary supplements marketed in Kosovo

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ABSTRACT

Water activity (aw) is a key physicochemical parameter governing moisture-driven processes in foods and dietary supplements and is widely used to characterize the availability of unbound water in complex matrices. Despite its relevance, market-based data on aw in commercial supplements are scarce, particularly for Kosovo. This study quantified aw in liquid, semi-solid, and solid dietary supplements and tested whether aw differs by dosage form and co-varies with moisture content and selected physicochemical parameters (pH, total dissolved solids (TDS), and electrical conductivity). Twenty products were collected from Kosovo retail markets and analyzed for water activity using a Rotronic HygroPalm HP23-AW-A meter (HC2-AW probe), for moisture content using Ohaus MB120/MB90 analyzers, and for additional physicochemical parameters using a calibrated multi-parameter meter. Liquid supplements exhibited higher aw values (0.509–0.951) than solid products (0.221–0.730), while the single semi-solid sample showed an intermediate value (0.616). Within the solid category, effervescent tablets consistently exhibited lower aw (0.221–0.320) than non-effervescent solids (0.419–0.730), indicating strong formulation-driven differences in unbound-water availability. The study identified statistically significant form-dependent differences in aw between liquid and solid supplements and demonstrated that, among solid formulations, effervescent matrices have lower aw than other solid forms. In solids, aw showed only a weak association with bulk moisture content, suggesting that matrix composition and processing can decouple aw from total moisture.

Keywords: dietary supplements, water activity, moisture content, physicochemical variability, Kosovo market.

INTRODUCTION

Water activity (aw) is a fundamental physicochemical descriptor of the energy state of water in a material and, unlike total moisture, reflects the fraction of water that is available to participate in phase transitions, dissolution, and moisture-driven chemical reactions. In dietary supplements, aw is influenced by formulation (e.g., sugars, polyols, salts), physical form, and processing, and it can therefore vary widely among products even when moisture contents appear similar (Scott, 1957; Karel, 1979; Labuza, 1980; Beuchat, 1987). Recent pharmacopeial and formulation-science literature has reinforced the value of measuring aw (in addition to moisture content) to better capture matrix–water interactions and storage-related stability behavior (United States Pharmacopeia, 2021; Modhave et al., 2024; Prada-Ramírez et al., 2024; Zäh et al., 2025). Because aw integrates solute effects and matrix binding, it is frequently used to interpret moisture-sensitive behavior such as caking, effervescence performance, and the propensity for hydrolytic degradation. Although aw can also constrain microbial growth in some matrices, physicochemical interpretation remains essential because preservative systems, pH, and formulation-specific factors modulate the consequences of a given aw value (Labuza, 1980; Slade et al., 1991; Chirife and Buera, 1994; United States Pharmacopeia, 2017; Snider et al., 2007).

From an environmental engineering and public-health perspective, baseline physicochemical characterization of widely consumed commercial products supports market surveillance and risk-informed decisions regarding storage conditions in humid climates and downstream waste handling. Dietary supplements are regulated as foods in many jurisdictions (e.g., the EU definition in Directive 2002/46/EC; European Parliament and Council, 2002). However, quantitative datasets that compare aw and related physicochemical parameters across dosage forms of dietary supplements are limited, especially in South-East Europe.

To address this gap for the Kosovo market, we analyzed 20 commercially available dietary supplements and determined water activity and moisture content in all samples, whereas pH, TDS, and electrical conductivity were assessed only in formulation types where these measurements were applicable. We tested three working hypotheses: (i) aw differs systematically between liquid and solid dosage forms; (ii) within solid products, processing type (effervescent vs non-effervescent) is associated with distinct aw ranges; and (iii) aw is not fully explained by bulk moisture content. The resulting dataset provides a regional baseline for comparative physicochemical assessment of supplements and for future, scope-aligned studies on moisture-driven processes.

MATERIALS AND METHODS

Sample collection, classification, and storage

A total of 20 dietary supplement products were purchased from the retail market in the Republic of Kosovo from multiple outlets and categorized by dosage form: 8 liquid, 1 semi-solid (soft), and 11 solid products. Products included water-soluble and fat-soluble vitamins, minerals, and mixed formulations. After purchase, all samples were stored at 20–22 °C in a clean, dry environment protected from direct light and excessive humidity, and were analyzed within 5 days to minimize storage-related changes.

Study workflow and data processing

To ensure reproducibility and clarify how the original results were obtained, the study was conducted according to the following standardized workflow:

1. Procurement and coding: purchase of commercially available products; documentation of label information and dosage form.
2. Controlled storage: standardized storage (20–22 °C; light/humidity protection) and fixed time-to-analysis (≤5 days).
3. Laboratory measurements: determination of water activity (aw) and moisture content/dry matter directly from the product; determination of pH, TDS, and electrical conductivity either directly (water-based liquids) or from prepared aqueous solutions/extracts (solids and oil-based products).
4. Data recording: raw readings recorded per sample and parameter, followed by unit verification and consistency checks.
5. Statistical processing: descriptive and inferential analyses performed to summarize results by dosage form and evaluate associations among measured parameters.

Instruments and materials

The following instruments and materials were used in this study:

- Analytical balance with a precision of 0.0001 g, Sartorius
- Water activity meter: Rotronic Hygropalm HP23-AW-A Sets
- Moisture and dry matter analyzer: Ohaus MB120 and MB90
- pH, TDS, and conductivity meter: Isolab
- Sample containers: PS-14.
- Sample holder: WP-40.
- HC2-*AW* detector.
- Aluminum trays for samples (90 mm diameter).

Determination of water activity (aw)

Water activity was measured using a portable Rotronic HygroPalm HP23-AW-A meter equipped with an HC2-AW probe. Measurements were performed at 20–22 °C (room temperature). Samples (1–2 g) were placed in PS-14 sample containers and positioned in the WP-40 holder. The HC2-AW detector was placed above the sample and readings were recorded after approximately 4–5 minutes (probe 1), once the display stabilized. Each sample was measured in duplicate, and the reported aw value represents the mean of the two measurements. Calibration was performed prior to measurements following the manufacturer's instructions (ISO, 2004). Measurement practices were also aligned with current pharmacopeial recommendations for water activity determination in nonsterile products (United States Pharmacopeia, 2021).

Determination of moisture content and dry matter

Moisture content and dry matter were determined using the Ohaus MB120 and MB90 moisture analyzer (Figure 1), which applies controlled halogen heating (40–200 °C) and provides a readability of 0.01%. In this study, a fixed drying program of 140 °C for 20 minutes was applied to all samples to ensure method consistency and comparability across different supplement forms. This temperature–time setting was selected as a practical compromise to achieve rapid moisture removal and stable final mass within a standardized measurement window. Moisture (%) and dry matter (%) were calculated by the instrument based on the mass loss during drying, with dry matter derived from the final stable mass.

Determination of pH, TDS, and electrical conductivity

The pH, TDS, and electrical conductivity of the samples were measured using a portable Isolab meter (Figure 2). After calibration, the probes were immersed in the samples until readings stabilized. Readings were considered stable when the display varied by less than 0.01 over 10 seconds. pH is reported in pH units; TDS in mg/L (ppm); and electrical conductivity in μS/cm. Solid samples were prepared as aqueous solutions by dissolving 10 g of sample in 50 g of distilled water. For liquid water-based supplements, pH, TDS, and conductivity were measured directly in the original liquid. For oil-based supplements, pH/TDS/conductivity are not directly applicable; therefore, these parameters were measured on an aqueous extract prepared by mixing 10 g of the product with 50 g of distilled water, followed by thorough mixing and separation, and measurements were taken from the aqueous phase. Probes and containers were rinsed with distilled water between measurements to avoid cross-contamination. Study limitations. This study did not include microbiological enumeration or time-based stability testing; therefore, interpretations are limited to physicochemical characterization and comparative analysis of aw and related parameters (ICMSF, 1980, 1996).

RESULTS AND DISCUSSION

Results are presented in the same order as the analytical workflow, from sample classification to water activity (*aw*), moisture content, pH, total dissolved solids (TDS), electrical conductivity, and the statistical analyses used for between-form comparisons.

Classification of analyzed supplements

A total of 20 dietary supplement samples were analyzed. Among these, 8 samples (40%) were in liquid form, 1 sample (5%) was semisolid (soft), and 11 samples (55%) were solid (Table 1, Figure 3).

Solid formulations constituted the majority of the analyzed products (55%), followed by liquids (40%) and one semisolid sample

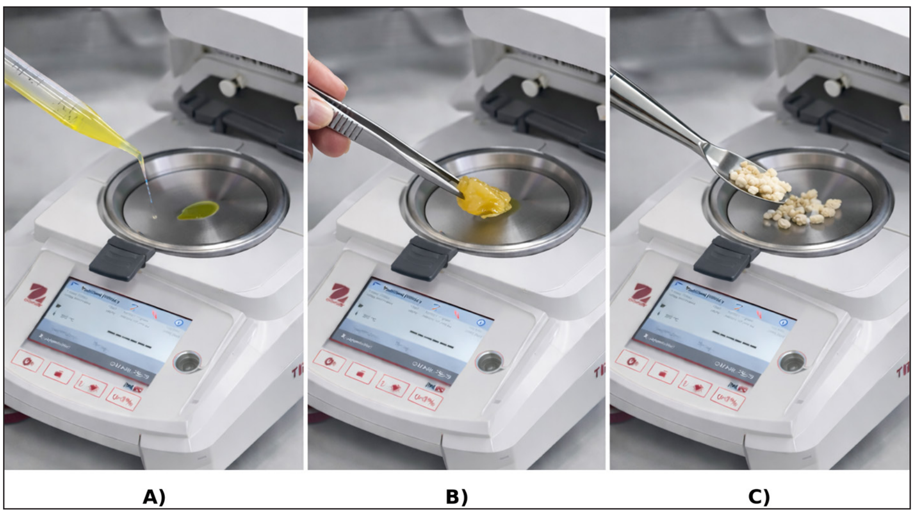

Figure 1. Determination of moisture content and dry matter using the Ohaus MB120 moisture analyzer during measurement of representative supplement matrices: (A) liquid, (B) semi-solid (soft), and (C) solid

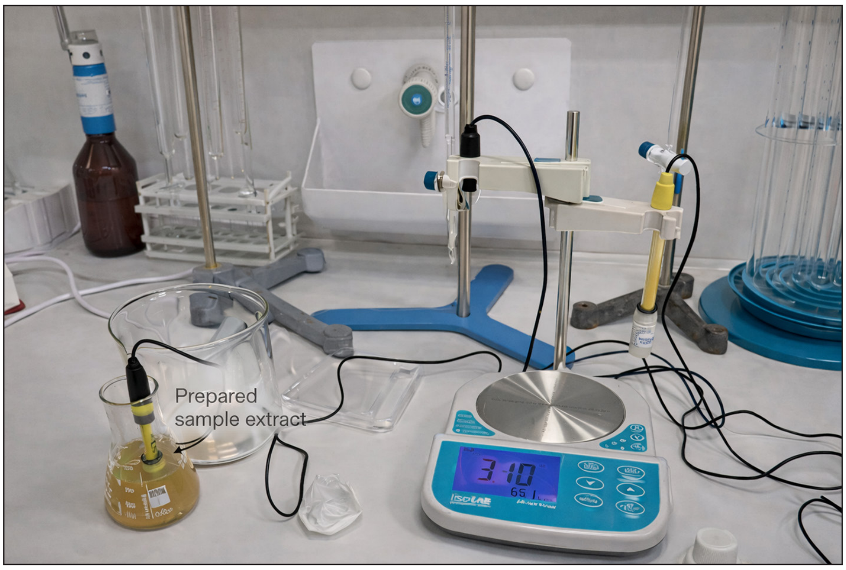

Figure 2. Multi-parameter analysis: pH, TDS, and electrical conductivity. The yellow liquid in the flask represents the aqueous extract of the dietary supplement sample used for measurement

Table 1. Classification of samples according to their physical consistency

Form type	Frequency	Percent	Valid percent	Cumulative percent
Liquid forms	8	40.0	40.0	40.0
Semi-solid (soft) forms	1	5.0	5.0	45.0
Solid forms	11	55.0	55.0	100.0
Total	20	100.0	100.0	

(5%). The predominance of solid dosage forms is consistent with common market practice, where tablets, capsules, and powders are widely used because they are convenient to package, transport, and handle and often exhibit lower free-water availability than many liquid formulations

Water activity in liquid forms

The water activity values of the liquid supplements were determined using the Rotronic Hygropalm portable device. The values obtained are presented below (Table 2 and Figure 4). Liquid supplements generally showed higher aw values than solid products, reflecting greater availability of unbound water in liquid matrices. Because aw is matrix- and solute-dependent, interpretation should consider formulation characteristics (e.g., sugars, polyols, oils) that can reduce effective water availability despite high total moisture (Scott, 1957; Labuza, 1980; Beuchat, 1987).

Water activity in semi-solid (soft) form

The single semi-solid supplement analyzed – coconut oil (soft) – had a water activity of 0.616 (Table 3). This intermediate aw value (0.616) indicates a moderate level of unbound water relative to the liquid and solid groups; however, because only one semi-solid product was analyzed, this observation should not be generalized.

Water activity in solid forms

The following table (Table 4) presents the measured water activity values for the solid forms of the analyzed dietary supplements. These values quantify unbound-water availability and provide a comparative indicator of formulation- and process-related differences within the solid category. Measured aw values for solid supplements ranged from 0.221 to 0.730 (ISO, 2004; Snider et al., 2007; United States Pharmacopeia, 2017) (Figure 5). The chart illustrates the measured water activity (*aw*) of selected solid-form

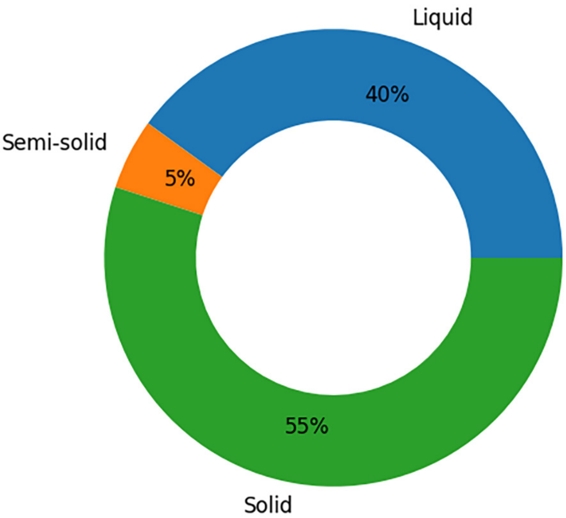

Figure 3. Classification of tested forms according to their physical consistency

Table 2. Water activity values in liquid forms

Sample	Water activity
Vitamin C (ampoule)	0.946
Memory Plus (oral suspension)	0.927
Vitamin D3 (oil)	0.625
Iron Energy (syrup)	0.921
Vitamin C (syrup)	0.786
Omega 3 (syrup)	0.951
Coconut oil (liquid)	0.509
Vitamin D3 (softgel)	0.739

Table 3. Water activity in semi-solid (soft) form

Sample	Water activity
Coconut oil (soft)	0.616

food supplements. The highest *aw* value was observed for Sulforaphane (caps.) (0.730), followed by Vit C + Zn (gran.) (0.702) and Vit C + Zn (caps.) (0.690). In contrast, the lowest *aw* value was recorded for Ca+Mg+Zn+D3+C (eff.) (0.221), followed by Vit C (eff.) (0.230) and Mg + E (eff.) (0.259). Overall, these results indicate notable variability in *aw* among solid supplements, likely reflecting differences in formulation and composition.

Moisture content

Moisture content was analyzed using the OHAUS MB120 and MB90 instrument (Table 5 and Figure 6). The results indicate that the highest moisture level was found in Sulforaphane capsules (13.9%), while the lowest was in Niacin tablets (0.86%).

pH values

pH values ranged from 1.35 to 7.22, depending on the formulation (Table 6). The results show that

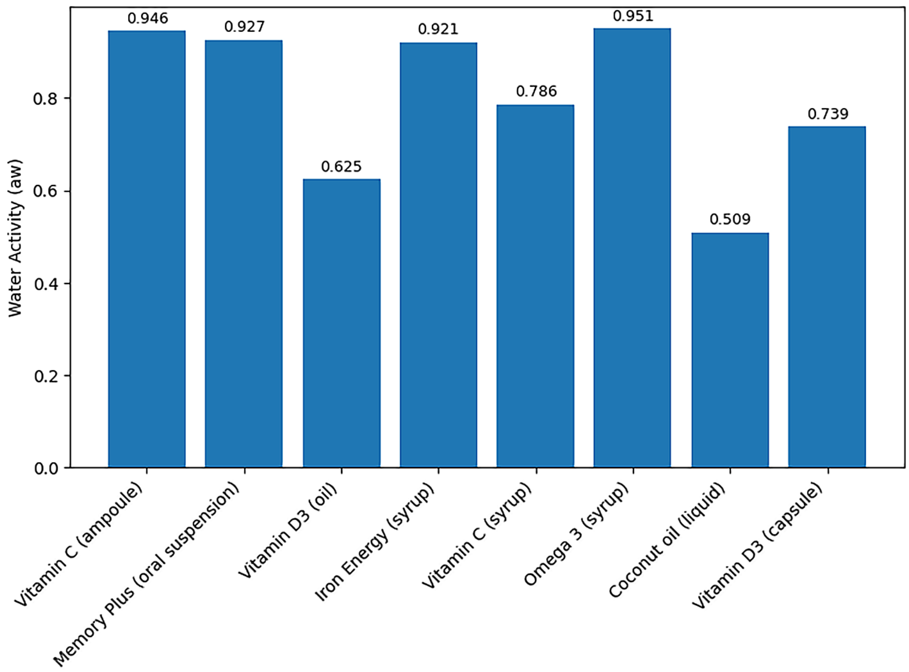

Figure 4. Water activity values in liquid supplements

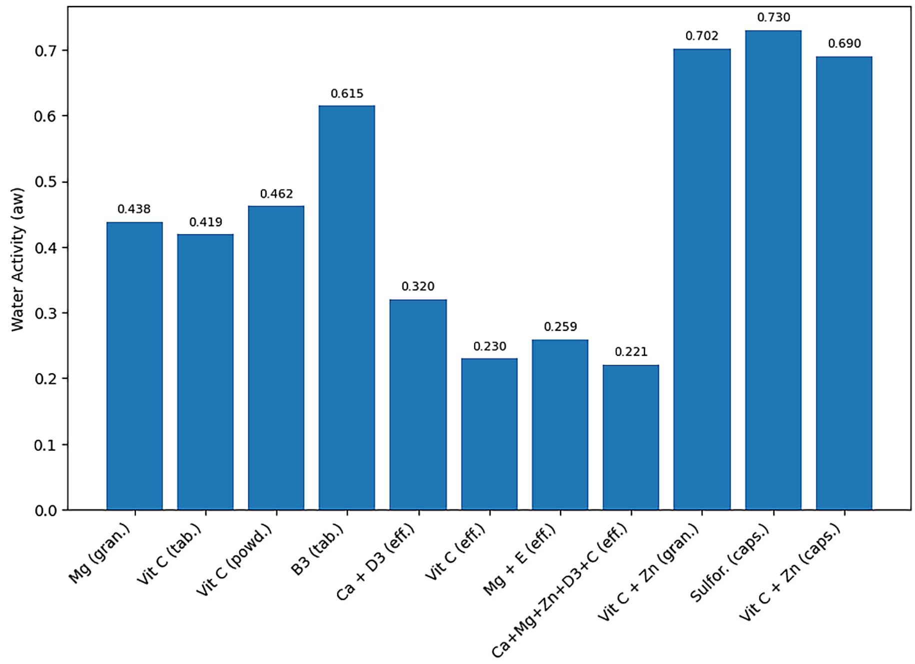

Figure 5. Water activity in solid forms

Table 4. Water activity values in solid forms

Sample	Water activity (aw)
Magnesium (granules)	0.438
Vitamin C (tablet)	0.419
Vitamin C (powder)	0.462
Niacin (Vitamin B3) (tablet)	0.615
Calcium + Vitamin D3 (effervescent)	0.320
Vitamin C (effervescent)	0.230
Magnesium + Vitamin E (effervescent)	0.259
Calcium + Magnesium + Zinc + Vitamin D3 + Vitamin C (effervescent)	0.221
Vitamin C + Zinc (granules)	0.702
Sulforaphane (capsule)	0.730
Vitamin C + Zinc (capsule)	0.690

Table 5. Moisture content (%) in analyzed samples

Sample	Moisture (%)
Magnesium (granules)	4.75
Vitamin C (tablet)	8.57
Vitamin C (powder)	9.83
Niacin (Vitamin B3) (tablet)	0.86
Calcium + Vitamin D3 (effervescent)	4.35
Vitamin C (effervescent)	10.83
Magnesium + Vitamin E (effervescent)	6.17
Calcium + Magnesium + Zinc + Vitamin D3 + Vitamin C (effervescent)	4.12
Vitamin C + Zinc (granules)	6.82
Vitamin D3 (capsule)	6.12
Sulforaphane (capsule)	13.90
Vitamin C + Zinc (capsule)	4.91

pH values ranged from 1.35 to 7.22, depending on formulation type and content. The wide pH range reflects differences in formulation composition and may influence dissolution behavior, ionic speciation, and moisture-driven chemical processes (Figure 7).

Total dissolved solids (TDS) and conductivity

TDS and conductivity values are summarized in Table 7. TDS and conductivity values reflect the ionic content of the samples, which can influence solubility, taste, and stability (Figure 8).

Statistical analysis of water activity measures

To support a rigorous interpretation of water activity (aw) measurements in dietary supplements, descriptive statistics were used to summarize aw levels across dosage forms.

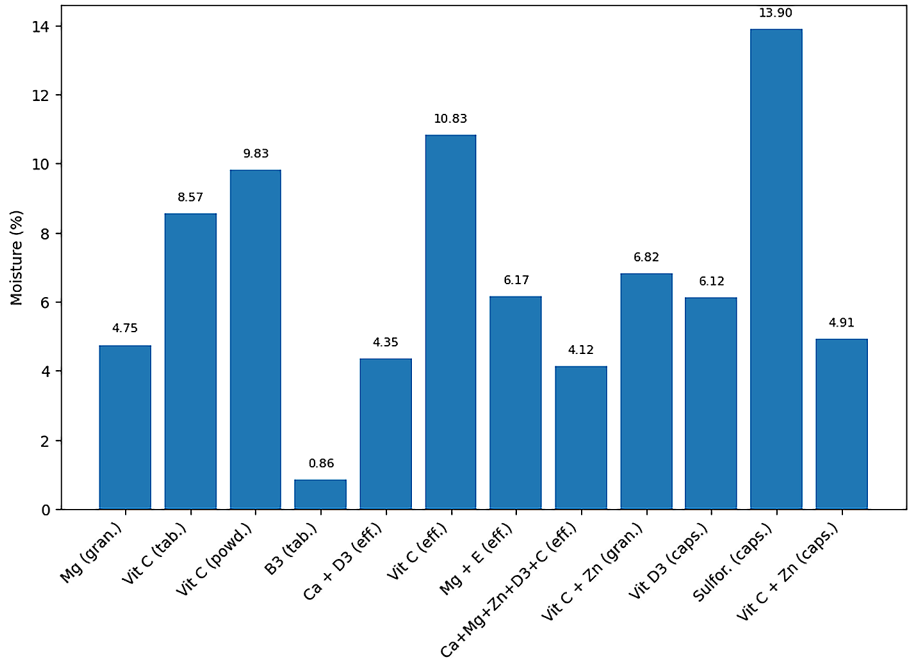

Figure 6. Moisture content in analyzed samples

Table 6. pH values of analyzed samples

Sample	pH
Magnesium (granules)	4.19
Vitamin C (tablet)	2.90
Vitamin C (powder)	1.87
Niacin (Vitamin B3) (tablet)	2.49
Calcium + Vitamin D3 (effervescent)	2.67
Vitamin C (effervescent)	3.55
Magnesium + Vitamin E (effervescent)	2.82
Calcium + Magnesium + Zinc + Vitamin D3 + Vitamin C (effervescent)	3.47
Vitamin C + Zinc (granules)	3.11
Vitamin C (ampoule)	7.11
Memory Plus (oral suspension)	2.61
Vitamin D3 (oil)	3.93
Iron Energy (syrup)	4.12
Vitamin C (syrup)	1.53
Omega 3 (syrup)	1.35
Coconut oil (liquid)	7.22
Sulforaphane (capsule)	5.71
Vitamin C + Zinc (capsule)	2.14

Inferential tests were then applied to evaluate (i) form-dependent differences in aw, (ii) within-solid differences between effervescent and non-effervescent matrices, and (iii) associations between aw and moisture content in solid products.

Table 7. TDS and conductivity values in analyzed samples

Sample	TDS	Conductivity
Magnesium (granules)	7.10	10.75
Calcium + Vitamin D3 (effervescent)	4.17	6.30
Vitamin C (effervescent)	14.9	22.4
Vitamin C (ampoule)	12.36	17.07
Memory Plus (oral suspension)	411	622
Vitamin C (syrup)	34.34	45.1
Omega 3 (syrup)	439	662
Sulforaphane (capsule)	415	640

Descriptive statistics by dosage form

Water activity values showed clear variability across formulations. Summary statistics (mean ± SD, min–max) were calculated separately for liquid, semi-solid (soft), and solid supplements to enable direct comparisons across forms (Table 8). Liquid samples showed the highest mean *aw* and a relatively wide range, while solid forms had a lower mean *aw* but also substantial variability. The semi-solid category included one product only; therefore, variability estimates (SD) could not be computed for this group. Liquid supplements generally exhibited higher *aw* values than solid products, whereas solid supplements

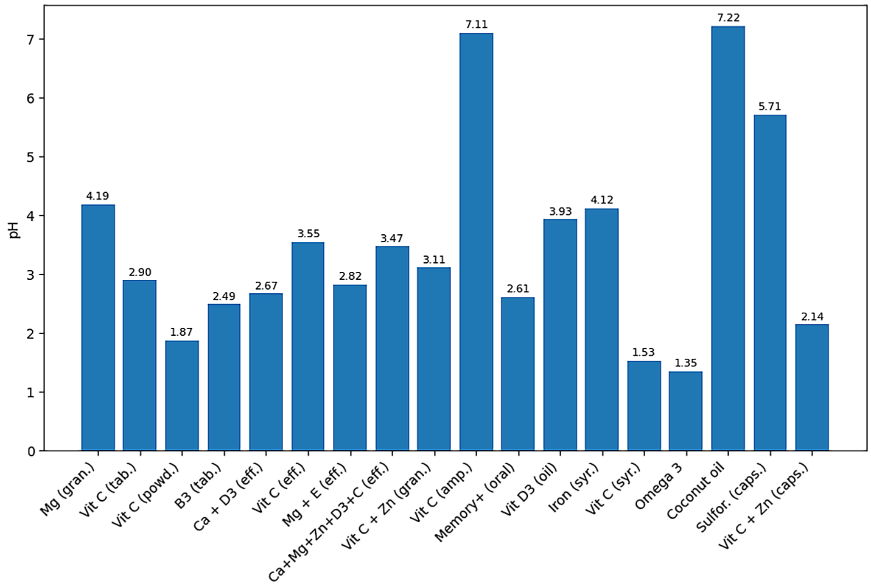

Figure 7. pH values of analyzed samples

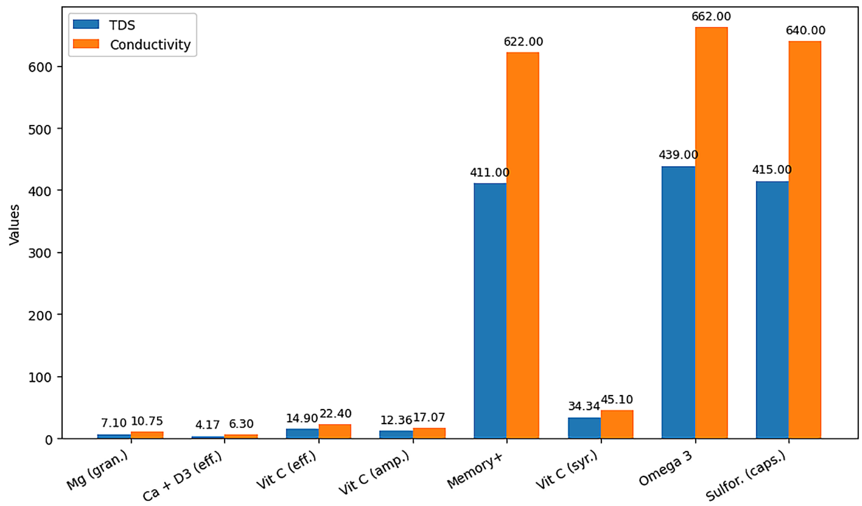

Figure 8. TDS and conductivity values

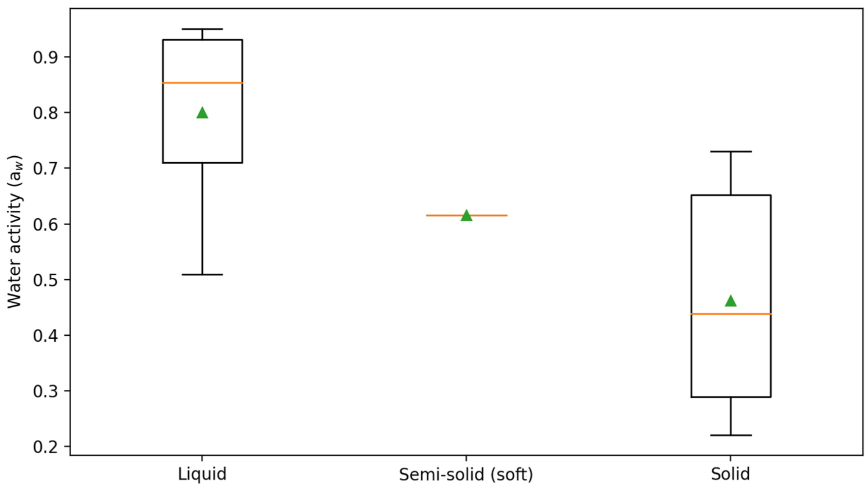

Figure 9. Water activity by dosage form (boxplot)

Table 8. Water activity summary statistics by dosage form (mean ± SD, min–max)

Dosage form	N	Water activity (mean ± SD)	Water activity (min–max)
Liquid	8	0.801 ± 0.167	0.509 – 0.951
Semi-solid (soft)	1	0.616	0.616 – 0.616
Solid	11	0.462 ± 0.195	0.221 – 0.730

showed lower central tendency but a wide spread, suggesting strong formulation-driven differences within the solid group (e.g., effervescent vs. tablets/capsules).

Visualization of aw patterns by form

Figure 9 summarizes the distribution of aw by dosage form using a boxplot. The plot highlights that liquid products cluster at higher aw levels overall, while solid products span a broader portion of the lower-to-moderate aw range. The semi-solid group appears as a single observation, reflecting the limited sample size in that category.

The figure provides two key insights. First, the liquid group is positioned well above the solid group, consistent with the descriptive means and ranges. Second, the solid group displays notable dispersion (0.221–0.730), indicating that "solid" form alone does not determine *aw*; rather, ingredient composition, hygroscopic excipients, and processing characteristics likely contribute to the observed variability. Because the semi-solid group includes only one sample, its graphical representation should be interpreted cautiously and not generalized.

Inferential testing and association analysis

- Between-form comparisons. Liquid supplements exhibited higher aw than solid supplements (median 0.854 vs 0.438; Mann–Whitney U = 81, p = 0.0012).
- Within-solid comparison. Effervescent tablets showed lower aw than other solid matrices (median 0.245 vs 0.615; U = 0, p = 0.0061), indicating that processing and formulation strongly influence unbound-water availability within solid products (Chatzidopavlaki et al., 2025; Veronica et al., 2022). Storage humidity and temperature can rapidly alter moisture distribution and physical stability in solid matrices through excipient-driven sorption/swelling phenomena, supporting the low-aw behavior observed for effervescent systems (Ibrahim et al., 2025).
- Association with moisture. Across solid products with matched measurements (n = 11), aw was weakly associated with moisture content (Spearman rho = 0.25, p = 0.47), suggesting that aw cannot be inferred from total moisture alone and is sensitive to solute composition and matrix binding (Table 9).

The combined descriptive and inferential results demonstrate pronounced formulation-driven heterogeneity in water activity across the Kosovo supplement market. Liquids cluster at higher water activity values, while solids span a wider low-to-moderate range; within solids, effervescent products form a distinctly low-aw subgroup. Together, these findings provide a scientifically substantiated baseline for comparative physicochemical assessment across dosage forms.

Moisture content, pH, TDS, and conductivity also varied across products and may contribute to moisture-driven behavior depending on composition and processing. Consistent with established water-activity theory, chemical reaction rates and physical transitions can change non-linearly across aw ranges, and solute systems may shift these responses (Karel, 1979; Labuza, 1980; Slade et al.,

Table 9. Summary the inferential tests and association analyses used in this study

Analysis	Variable / Comparison	N	Group statistic	SD	SE	Test statistic	p (2-tailed)
Mann–Whitney U	aw: liquids vs solids	19	Median_liq=0.854; Median_sol=0.438	—	—	U=81	0.0012
Mann–Whitney U	aw in solids: effervescent vs non-effervescent	11 (4 vs 7)	Median_eff=0.245; Median_non=0.615	—	—	U=0	0.0061
Spearman correlation	Solids: aw vs moisture content (%)	11	—	—	—	rho=0.25	0.47

Note: U = Mann–Whitney U statistic; rho = Spearman rank correlation; p = two-tailed p-value.

1991; Chirife and Buera, 1994; Beuchat, 1987). The weak water activity – moisture association observed in solid products highlights the need to interpret aw as a matrix property rather than a proxy for total moisture (Scott, 1957; Snider et al., 2007). Similar market-level variability in moisture-related attributes of dietary supplements (and the role of packaging/moisture ingress) has also been reported elsewhere, underscoring the need for region-specific datasets (Amidžić Klarić et al., 2023).

CONCLUSIONS

This study achieved its objective by producing the first market-based dataset of water activity (aw) and selected physicochemical parameters for dietary supplements marketed in Kosovo, thereby establishing a region-specific baseline for comparative assessment across dosage forms. The study delivers new, scientifically substantiated results based on non-parametric testing:

- aw differs significantly by dosage form, with liquid supplements exhibiting higher aw than solid products (Mann-Whitney $U = 81$, $p = 0.0012$).
- within solid supplements, effervescent tablets form a distinct low-aw subgroup compared with other solid matrices ($U = 0$, $p = 0.0061$).
- in solid products, aw is only weakly associated with bulk moisture content (Spearman rho $= 0.25$, $p = 0.47$), indicating that matrix/formulation effects are key drivers of unbound-water availability.

These findings fill a regional knowledge gap by quantifying real-market variability in aw and demonstrating that dosage form and solid-matrix type are principal determinants of moisture-state behavior in commercially available supplements in Kosovo. Building on this baseline, future scope-aligned studies can quantify moisture-driven physicochemical changes under realistic storage and environmental exposures (e.g., seasonal relative humidity and temperature variability, storage duration, and packaging integrity), and evaluate packaging- and handling-related moisture ingress as determinants of aw dynamics in marketed products. Where appropriate, integrated designs may additionally include targeted microbiological enumeration to directly test whether water activity and moisture-related indicators translate into measurable microbial outcomes under real-world storage conditions.

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