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Article

Characterization of the Chemical Composition of Sediments and the Presence of Thermotolerant Coliforms in Different Sub-Basins of the Rio Grande

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ABSTRACT

Natural and anthropogenic factors influence water and sediment in springs. In rural areas, agricultural and livestock activities affect the dynamics and structure of spring ecosystems. This study aimed to assess differences in the sediment chemical compositions and the presence of thermotolerant coliforms in the water of fifteen springs, in Serrinha region, located in rural landscape of the five Grande River's sub basins. Concentrations of metals and nutrients were analyzed, along with density estimates of thermotolerant coliforms, to correlate these parameters with land use and occupation. Lead, chromium, nickel, and manganese were not detected in the samples, considering the detection limits of the analytical equipment. Principal component analysis (PCA) indicated that sub-basin 3 had the highest calcium concentrations, while sub-basin 6 exhibited elevated levels of phosphorus, aluminum, and magnesium. Sub-basin 2 had high concentrations of potassium and iron, while sub-basins 1 and 4 showed the highest levels of copper and thermotolerant coliforms, which are probably associated with pasture areas around springs. The coliforms values in these sub-basins suggest a direct influence of livestock activities, emphasizing the need for management practices that prevent the animal waste contact with spring water. The results indicate that the dynamics of chemical elements in sediments are directly related to land use in the region. These findings underscore the importance of adequate preservation of riparian vegetation and controlling diffuse pollution to ensure water resource quality.

Keywords: brazilian savanna; first-order stream; spring contamination; toxic metals; fecal coliforms.

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RESUMO
A água e os sedimentos em nascentes são influenciados por fatores naturais e antrópicos. Em áreas rurais, a atividade agropecuária exerce influência na dinâmica e estrutura dos ecossistemas de nascentes. Este estudo teve como objetivo verificar diferenças na composição química dos sedimentos e na presença de coliformes termotolerantes na água de 15 nascentes da região da Serrinha, localizadas em paisagem rural de cinco sub-bacias do Rio Grande, no Cerrado Mineiro. Foram analisadas concentrações de metais e nutrientes, além da estimativa da densidade de coliformes termotolerantes, a fim de correlacionar esses parâmetros com o uso e ocupação da terra. Os elementos chumbo, cromo, níquel e manganês não foram detectados nas amostras, dentro dos limites do equipamento utilizado. A análise de componentes principais (ACP) indicou que a sub-bacia 3 apresentou maiores concentrações de cálcio, enquanto a sub-bacia 6 se destacou pelo teor elevado de fósforo, alumínio e magnésio. A sub-bacia 2 apresentou altos níveis de potássio e ferro, enquanto as sub-bacias 1 e 4 mostraram as maiores concentrações de cobre e coliformes termotolerantes, possivelmente associadas à presença de áreas de pastagem próximas. Os valores de coliformes nestas sub-bacias sugerem influência direta da atividade pecuária, reforçando a necessidade de práticas de manejo que impeçam o contato de dejetos animais com a água das nascentes. Os resultados indicam que a dinâmica dos elementos químicos nos sedimentos está diretamente relacionada ao uso da terra na região. Esses achados reforçam a importância da preservação adequada da vegetação ciliar e do controle da poluição difusa para garantir a qualidade dos recursos hídricos.
Palavras-chave:. cerrado, rios de primeira ordem; contaminação de nascentes; metais tóxicos; coliformes fecais.

Introduction

The Brazilian Cerrado is one of the largest tropical savannas in the world in terms of area, and, as a result of its size, it has become an attractive region for economically driven cultivation (Pinto 2020). Despite high levels of aluminum and naturally low concentrations of calcium and magnesium in the soil, plant biodiversity is remarkably high in dystrophic Cerrado areas (Haridasan 2008). This diversity highlights the ecological importance of the biome, which is considered a global hotspot and the most impacted among all Brazilian biomes (Reis 2022). The biome is under intense anthropogenic pressure, with land conflicts and high deforestation rates that have been increasing since the 1990s (Silva et al. 2023). Its climate is marked by two distinct seasons: a dry winter and a rainy summer. Concentrated rainfall influences soil processes, leaching nutrients and degrading soil quality, with direct consequences for hydrology (Nascimento & Novais 2020).

Six of Brazil's eight major hydrographic basins have their headwaters in this region: the Amazon basin (Xingu, Madeira, and Trombetas rivers), the Tocantins basin (Araguaia and Tocantins rivers), the North/Northeast Atlantic basin (Parnaíba and Itapecuru rivers), the São Francisco basin (São Francisco, Pará, Paraopeba, das Velhas, Jequitaí, Paracatu, Urucuia, Carinhanha, Corrente, and Grande rivers), the East Atlantic basin (Pardo and Jequitinhonha rivers), and the Paraná/Paraguay basin (Felfili et al. 2005).

Springs represent points of groundwater emergence, and riparian vegetation plays a key role in their preservation and ecological stability. According to Law No. 12,651/2012 (Brasil 2012), these natural environments are responsible for the formation of watercourses, which, in their early stages, are classified as first-order streams based on flow and volume. These streams can merge to form higher-order rivers. This process underscores the importance of conserving springs to ensure the integrity of hydrographic networks and, consequently, the biodiversity associated with various biomes (Moreira et al. 2019). Springs also act as natural filters for rainwater, gradually releasing it to the earth's surface (Anyanwu & Onyele 2018). Moreover, they contribute to the transport and redistribution of organic matter in aquatic ecosystems. Physical and chemical changes caused by spring water, such as variations in turbidity and oxygenation, act as important ecological stimuli for the reproduction of several fish species (Carvalho & Uieda 2004).

In this regard, the Cerrado stands out as a strategic region for the maintenance of Brazil's hydrological systems, due to its central location and high altitude. This geographical configuration promotes the so-called "umbrella effect," a term describing the dispersal of surface waters in multiple directions from the central plateau, feeding various adjacent basins (Souza 2019). As a consequence of this peculiar relief and intense leaching, Cerrado soils are naturally nutrient-poor, which promotes sediment transport to regional water bodies.

This process directly affects the physical and chemical composition of spring sediments, interfering with water quality and related ecological processes (Novais 2019).

Sediments are integral components of aquatic ecosystems, defined as suspended or deposited solids, of mineral or organic origin, that serve as the main matrix transported by water (Owens 2008). They can be used to monitor the impact of anthropogenic pollution, such as contamination by toxic metals (Alves et al. 2014).

Toxic metals are elements that are not part of organisms' biological cycles and whose presence interferes with biochemical or physiological processes, with some showing chemical affinity with DNA and promoting carcinogenic responses (Garcia 2022). Metallic pollutants are persistent, undergo biogeochemical recycling, and pose environmental risks. Sediments have the capacity to absorb trace metals from the water and are associated with metallic species, depending on the geological characteristics of the drainage basin (Paula-Filho et al. 2015).

This study aims to evaluate the variation in the chemical composition of sediments and the concentration of thermotolerant coliforms in water among different hydrographic sub-basins located in a Cerrado region.

Materials and Methods

Study Area

The study was conducted in the region known as *Serrinha*, in the municipality of Frutal, Minas Gerais, between latitudes 19°46'52"–20°01'28" S and longitudes 48°49'29"–49°08'53" W (Figure 1). The average annual rainfall in the region is approximately 1,600 mm, with a mean annual temperature of 24°C. The climate is classified as *Aw* (tropical with a dry and cool winter season, and higher rainfall in summer; Alvares et al. 2014). This climate, combined with the geomorphological structure, defines the *Cerrado Phytogeographic and Morphoclimatic Domain*, where the study area is located.

Serrinha forms the watershed divide between the Paranaíba and Grande rivers, composed of arenitic rocks from the Uberaba Formation (Bauru Group), followed to the south by a band of the Vale do Rio do Peixe Formation (CPRM 2012). The edges of this formation comprise a critical recharge area for maintaining groundwater quality and quantity, as well as its outcrops. Recently, 702 springs were identified in the basins of the main streams forming the hydrographic network of Frutal: *Ribeirão São Mateus, Ribeirão Marimbondo, Córrego Bebedouro, Ribeirão Frutal*, and *Rio São Francisco* (Panarelli et al. 2025). All these streams originate in the southern portion of *Serrinha*, forming sub-basins of the Rio Grande watershed.

Protecting *Serrinha* is essential to prevent deforestation, improper land use, and the establishment of potentially polluting activities (Brasil, 2007) in an environment vital for water resource conservation, where agribusiness generates significant financial resources (IBGE 2023).

Sampling point and locations

Figure 1 shows the study area, highlighting the headwaters of the Rio Grande sub-basins, which include the studied spring zones. In five hydrographic sub-basins (1, 2, 3, 4, 6), three first-order river stretches were selected, with three sediment replicates collected per stretch (Figure 1) during dry and rainy seasons, totaling 90 samples. Sub-basin 5 was excluded due to inaccessible springs.

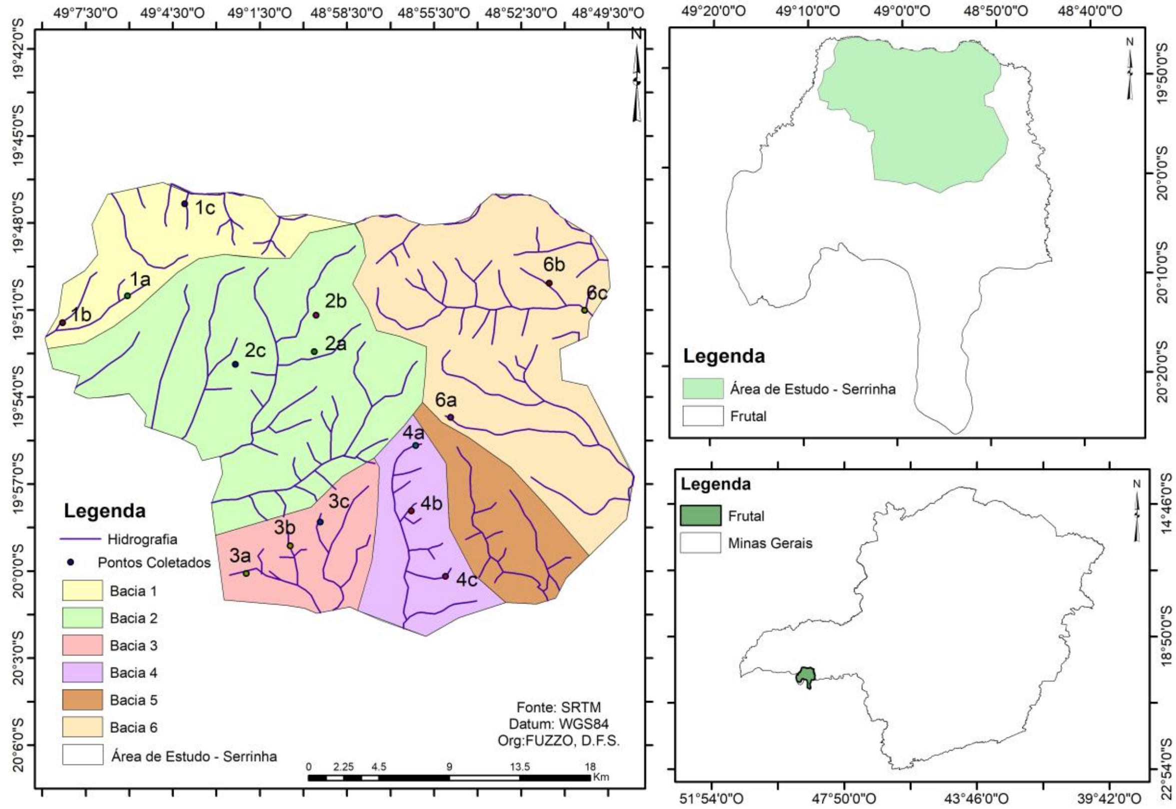

Figura 1. Location of the study area within the sub-basins of the principal rivers in Frutal municipality, Minas Gerais, Brazil: 1 – Ribeirão São Mateus; 2 – Ribeirão Marimbondo; 3 – Córrego Bebedouro; 4 – Ribeirão Frutal; 5 - Córrego São Bento da Ressaca (brown area – not sampled); 6 – São Francisco. Organização: D.F.S. Fuzzo, 2024

Figure 2 shows the altimetry of the study area. The Serrinha region in Frutal (MG) features elevations ranging from <530 m to >660 m. The lowest portions, located in the south and southwest, are concentrated in valley bottoms, while the highest elevations in the north-central area correspond to watershed divides. This altimetric variation directly influences surface runoff, land use, and erosion potential. Lower areas are typically used for agriculture and pasture, while higher elevation zones require more careful management practices.

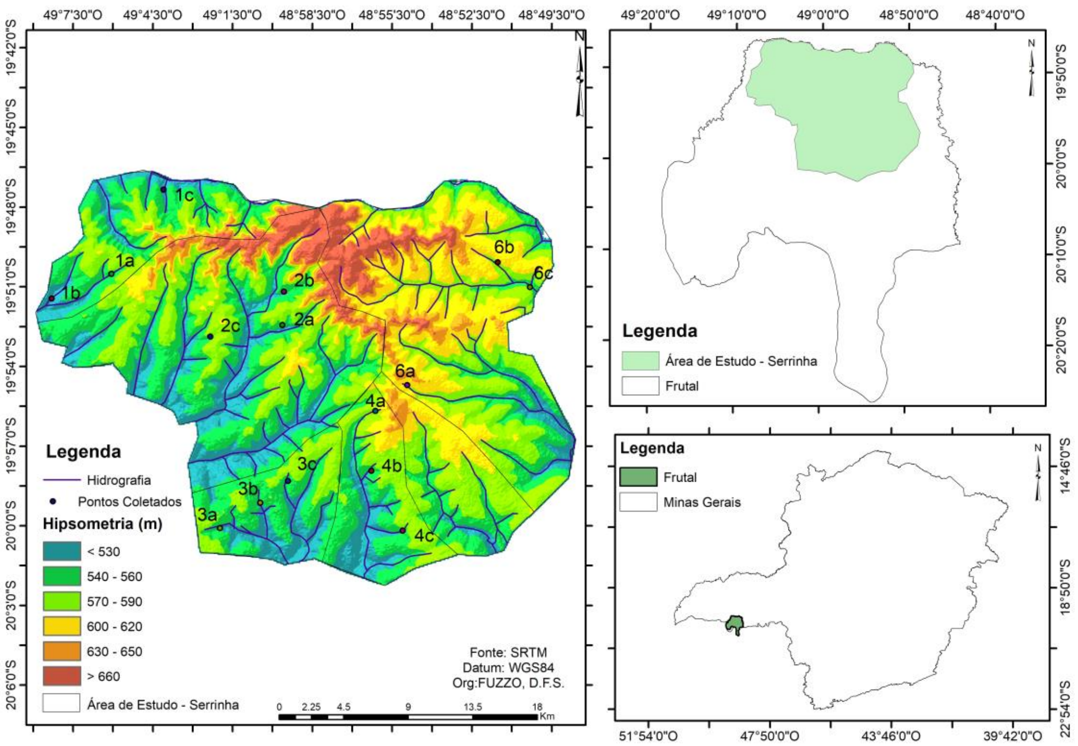

Figure 2. Altimetry of the Serrinha region and the points selected for analysis. Organization: D.F.S. Fuzzo, 2024

Sediment samples were collected during both dry and rainy seasons in 2023 using a 50-mm diameter PVC tube. Samples were stored in properly labeled plastic containers and immediately frozen until laboratory processing.

Sediment Analysis

Samples received at the laboratory were recorded in an Excel spreadsheet for traceability. The analytical procedures for quantifying target elements included six stages: drying, weighing, sample digestion, dilution (when required), equipment reading, and data entry.

Samples were oven-dried at 45°C for 12 hours, then sieved through a 20-mesh screen to remove branches, stones, and other unwanted material. Weighing was performed using a semi-analytical balance to obtain 5 g of fine oven-dried earth (FODE), which was transferred to 80-mL polyethylene bottles. Subsequently, 50 mL of extractant solution was added (1:10 ratio). Bottles were shaken at 200 rpm for 5 minutes on an orbital shaker, then left to settle for 16 hours. This procedure allowed particulate matter to settle, enabling collection of a supernatant aliquot for analysis. For calcium and magnesium determination, 0.5 mol/L strontium chloride ($SrCl_2$) was added as a flame stabilizer for atomic absorption spectrophotometry (AAS). Phosphorus analysis required a chromogenic solution containing ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$), ascorbic acid ($C_6H_8O_6$), sulfuric acid ($H_2SO_4$), and bismuth subcarbonate ($(BiO)_2(CO_3)$).

For analysis of lead (Pb), chromium (Cr), nickel (Ni), manganese (Mn), copper (Cu), zinc (Zn), iron (Fe), calcium (Ca), and magnesium (Mg), a Perkin Elmer Model 3110 atomic absorption spectrophotometer was used. Potassium (K) was measured using a flame photometer (Micronal). For phosphorus (P) analysis, a UV-Vis spectrophotometer (Analytik Jena) was employed. Aluminum (Al) was quantified by titration. All samples were processed following the extraction methodology recommended by EMBRAPA (2017). For evaluation of calcium, magnesium and aluminum, a 1.0 mol/L KCl solution was used at a 1:10 sediment-to-solution ratio.

For analysis of lead, chromium, nickel, manganese, copper, zinc, iron, phosphorus and potassium, the extracting solution was Mehlich-1 (0.05 mol/L HCl + 0.0125 mol/L H_2SO_4) (Mehlich 1953) at a 1:10 sediment-to-solution ratio. Aluminum was titrated with 0.025 mol/L NaOH using 1.0 mol/L bromothymol blue as indicator, until color change from yellow to blue was observed.

Calibration curves were prepared for each element analyzed by equipment following Lambert-Beer's Law, where absorbances of standard solutions with known concentrations were measured and the linear equation $y = ax + b$ was calculated. Only curves with $r^2 > 0.9800$ were accepted. All dilutions were accounted for to obtain the actual concentrations of elements detected in the sediments. Table 1 presents the quantification limits for each analyzed chemical element.

Table 1. Quantification limit of the chemical elements analyzed

Element	Quantification limit (mg L^{-1})
Ca	0,003
Mg	0,002
Zn	0,001
Cu	0,003
K	0,632
P	0,029
Fe	0,500
Al	0,010
Pb	0,020
Cr	0,250
Ni	0,030

Source: compiled by the authors.

Thermotolerant Coliforms

Water sampling for thermotolerant coliform determination was conducted using 500 mL pre-sterilized glass bottles. Quantification employed the Colilert test kit, where sample dilutions (1 mL, 0.1 mL, and 0.01 mL) were added to culture medium. Samples were incubated in Quanti-Tray/2000 plates for 24 hours at 35°C ± 0.5°C. Results were interpreted using the MPN/100 mL table (IDEXX 2017).

Land Use/Land Cover Mapping

RGB-432 color composite maps were generated using Landsat 8/OLI satellite images provided by NASA through the United States Geological Survey (USGS) website. A cartographic base map with watershed boundaries and drainage networks was created from SRTM (Shuttle Radar Topography Mission) data, a collaborative effort of the American (NASA and NGA), Italian (ASI), and German (DLR) space missions, with 30 m spatial resolution. Data were processed in a GIS environment using QGIS software (version 3.16), and watershed delineation was performed using the GRASS plugin, where elevation raster data were created to identify depressions and derive sub-basins. The watershed boundary was determined based on the outlet point (control section). The resulting file was converted to vector format for subsequent drainage network analysis. Municipal boundary vector images were obtained from the IBGE (Brazilian Institute of Geography and Statistics) website.

For land use and land cover identification and analysis, images from the MapBiomas catalog were used, available free of charge at https://brasil.mapbiomas.org/. These are raster format maps (30×30 m pixels) based on Landsat 5/TM and 8/OLI collections from 1985 to 2023. The platform data are automatically classified and cloud-processed to generate an annual historical series of land cover and land use maps of Brazil (Moraes 2020). Each image pixel is classified into one of 27 land-use categories (MAPBIOMAS 2019). The data were processed using QGIS 3.16 software, enabling spatial quantification analysis of land use.

Statistical analysis of data

A principal component analysis (PCA) was performed to compare the chemical composition of sediments and thermotolerant coliforms in water among the sub-basins. The data were standardized to homogenize variances (Legendre & Legendre 2012). Analyses were conducted using Statistica 14 software (Cloud Software Group 2023).

Results and discussion

The elements lead, chromium, nickel and manganese were not detected by the quantitative method used in this study, which does not indicate the absence of these chemical species in the samples, only that, if present, they were below the detection limit of the equipment used (Table 1).

The first factorial plane of the principal component analysis involving the hydrological sub-basins of the Serrinha region retained 71.09% of the original data variability (PC1 = 38.27% and PC2 = 32.82%). Sub-basin 3 (B3) was positioned on the positive side of principal component 1, associated with calcium. In contrast, sub-basin 6 was positioned on the negative side of principal component 1, associated with the variables total phosphorus, aluminum and magnesium. Sub-basin 2 was positioned on the positive side of principal component 2, associated with the variables potassium and iron. In contrast to this pattern, sub-basins 1 and 4 were positioned on the negative side of principal component 2, being associated with thermotolerant coliforms and copper (Figure 3).

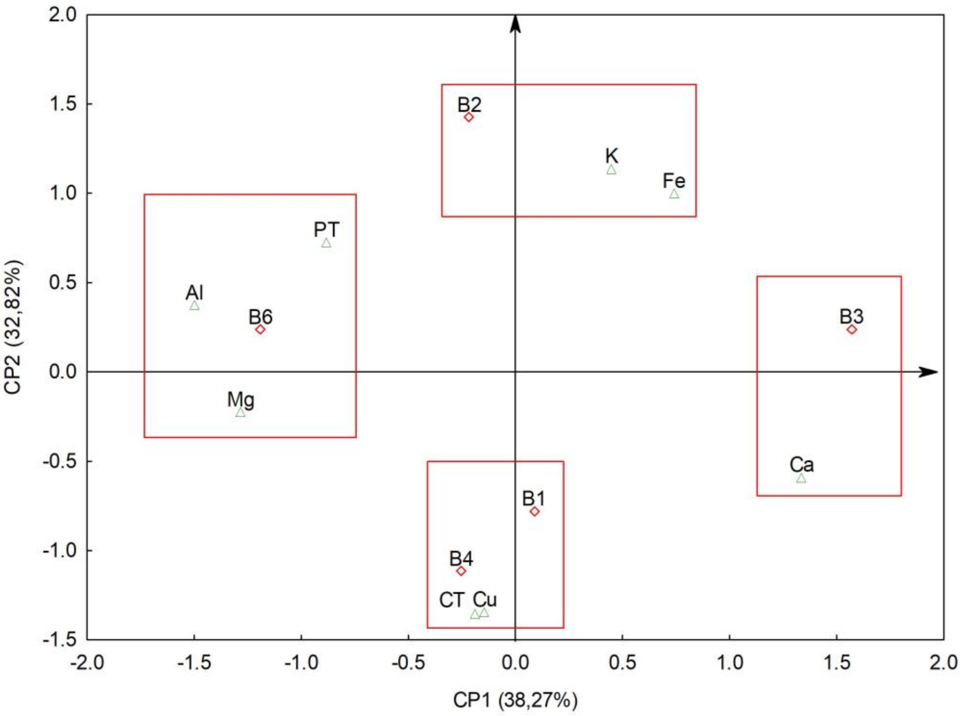

Figure 3. Biplot graph of the principal component analysis involving the sampled hydrographic sub-basins in the Serrinha region of Frutal municipality, Minas Gerais, disregarding the influence of dry and rainy periods, where: B1 to B6 = hydrographic sub-basins; CT = thermotolerant coliforms; K = potassium; Al = aluminum; Mg = magnesium; Ca = calcium; Fe = iron; PT = total phosphorus. Source: Authors (2025).

Sub-basin 3 showed the highest mean calcium values (13.3 mg L^{-1}), while sub-basin 2 showed the lowest (3.1 mg L^{-1}). Sub-basin 6 had the highest mean concentrations of total phosphorus (9.0 mg L^{-1}), aluminum (87.7 mg L^{-1}), and magnesium (2.1 mg L^{-1}). Sub-basin 2 exhibited the highest mean levels of potassium (K, 21.1 mg L^{-1}) and iron (Fe, 64.2 mg L^{-1}) (Table 2). Sub-basins 1 and 4 recorded the highest thermotolerant coliform counts (515.1 and 1312.1 MPN 100 mL^{-1}, respectively) and copper concentrations (0.09 and 0.08 mg L^{-1}, respectively).

Table 2. Mean and standard deviation of quantified chemical elements (mg L^{-1}) and thermotolerant coliforms (TC - MPN 100 mL^{-1}) in the hydrographic sub-basins (B1-B6).

Sub-basins	PT	K	Ca	Mg	Al	Fe	Cu	CT
B1	3,9 ±3,4	16,1 ±6,7	7,0 ±6,7	2,1 ±0,9	75,5 ±20,9	45,5 ±18,5	0,09 ±0,22	515,1 ±576,9
B2	5,9 ±5,3	21,1 ±9,0	3,1 ±9,5	2,2 ±1,0	82,4 ±17,6	64,2 ±23,2	0,05 ±0,03	124,9 ±121,3
B3	5,3 ±5,6	18,4 ±6,3	13,3 ±14,4	1,3 ±0,9	63,6 ±18,6	47,0 ±34,2	0,05 ±0,11	324,0 ±416,5
B4	4,7 ±3,9	17,6 ±6,1	7,9 ±7,1	2,3 ±0,7	77,0 ±22,6	22,4 ±13,8	0,08 ±0,12	1312,1 ±3415,8
B6	9,0 ±12,8	15,8 ±5,9	4,2 ±4,9	2,1 ±1,5	87,7 ±24,8	18,5 ±14,6	0,06 ±0,02	356,6 ±581,0

Source: Authors (2025).

In sub-basins 1 and 4, statistical analysis indicated high quantities of thermotolerant coliforms - bacteria found in fecal matter from homeothermic animals (Brito et al. 2024). All sampled springs are at least partially located within pasture areas (Figure 4; Table 3), with two springs in sub-basin 1 showing altered riparian forest conditions, while springs in sub-basin 4 maintain relatively preserved riparian vegetation (according to António et al. 2023). However, thermotolerant coliform levels presented alarming values, indicating that even with existing vegetation, the springs remain unprotected from cattle access or manure runoff. Bovine feces were observed in the region, associated with pastureland, demonstrating that animal proximity and land cover type promote fecal contamination reaching these points, and that the area should be protected and inaccessible to livestock (Santos et al. 2021).

Copper showed higher concentrations in sub-basins 1 and 4, though values remained below the maximum limit of 2.0 mg L^{-1} established by CONAMA 357/2005 (Brazil 2005) for water (no reference exists for sediment). Despite being within regulatory limits, increased copper levels were observed in areas adjacent to pastures and sugarcane fields, with leaching of agricultural chemicals used in cultivation practices into the water system, ultimately reaching the sediment (Moreira et al. 2024).

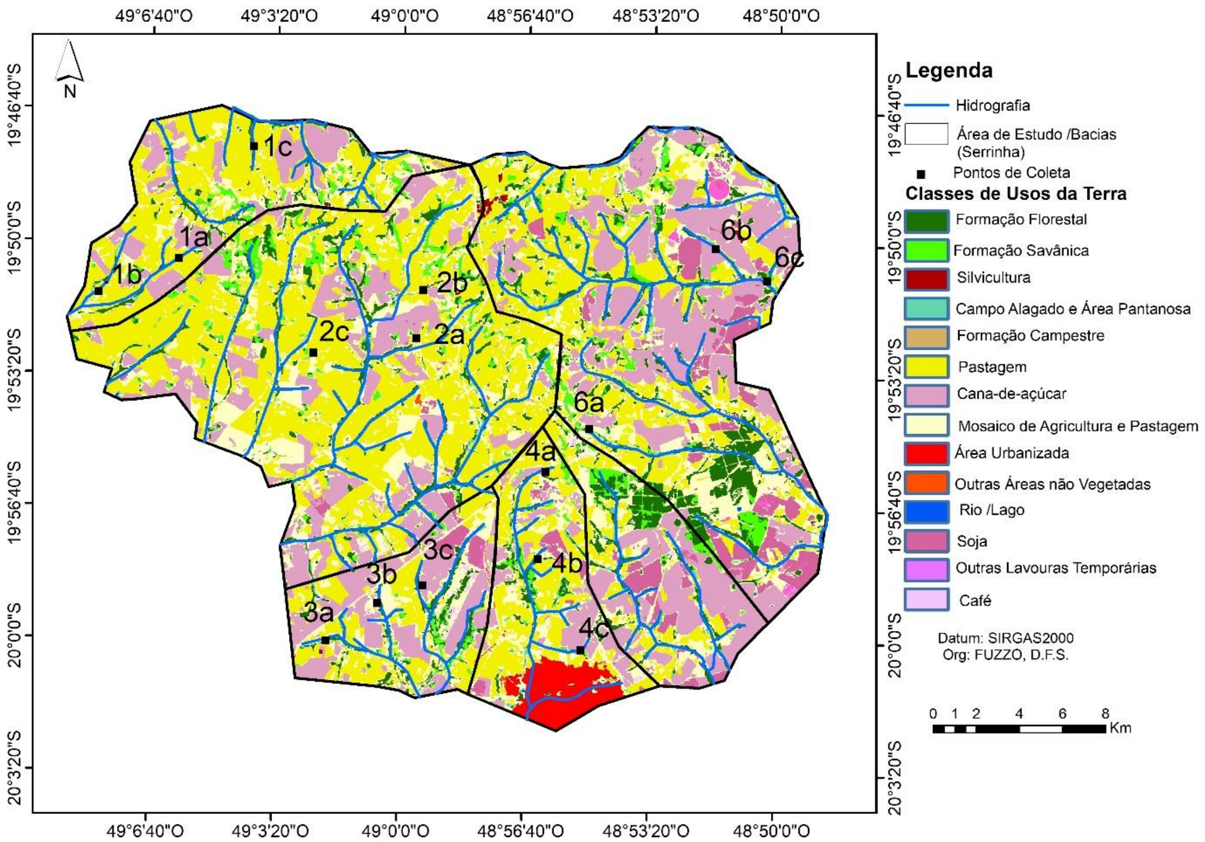

Figure 4. Land use and land cover in the Serrinha region, Frutal municipality, Minas Gerais, where: 1 = São Mateus stream sub-basin; 2 = Marimbondo stream; 3 = São José do Bebedouro creek; 4 = Frutal stream; 6 = São Francisco river. Organization: D.F.S. Fuzzo, 2024.

Table 3. Association between PCA results and predominant land use/land cover in each sub-basin.

Sub-basin	Parameter grouped by PCA	Allochthonous environment
B1	Thermotolerant coliforms and copper	Pasture
B2	Potassium and Iron	B2A & B2B: Pasture and sugarcane B2C: Pasture
B3	Calcium	Pasture and sugarcane
B4	Thermotolerant coliforms and copper	B4A: Pasture and sugarcane B4B: Pasture B4C: Pasture and sugarcane
B6	Aluminum, magnesium and total phosphorus	B6A: Pasture B6B: Sugarcane B6C: Pasture and sugarcane

Source: Authors (2025).

In sub-basin 2, potassium and iron were found in higher quantities. In this sub-basin, pasture and sugarcane predominated, with a well-distributed drainage network (Figure 4; Table 3). Potassium is an important nutrient for top-dressing fertilization in pasture (Nascimento 2024). Potassium has high soil mobility and is easily leached by rain (Medeiros et al. 2021), which may explain the presence of this element in spring sediments, especially in sub-basin 2. Iron is an element of great abundance in Cerrado soils and has low mobility in soil and sediment, being present even in agricultural soils of this biome (Botrel et al. 2020).

In sub-basin 3, the presence of pasture and sugarcane is observed (Figure 4), associated with the calcium element (Table 3), which is more present. A reduced amount of aluminum is also noted. Thus, such concentrations may be associated with liming activities for soil preparation aimed at sugarcane cultivation. Cerrado soils are characterized by high aluminum concentrations, which is why annual correction is necessary to make nutrients available to plants in arable areas.

Sub-basin 6 is associated with sugarcane cultivation, pasture, and forest formation (Figure 4; Table 3), and the elements phosphorus, aluminum, and magnesium were associated with this basin. The presence of aluminum demonstrates that the sediment is characterized according to the biome. Magnesium and phosphorus are elements found in agricultural fertilizers; the macronutrient magnesium can be applied via soil through the liming process or foliarly. Agricultural lime has the power to neutralize aluminum, therefore, it can be inferred that magnesium was applied foliarly, since aluminum was present in high concentrations. Phosphate fertilization is necessary for the primary growth of sugarcane, with this element being provided through soil fertilization (Garcia & Mendes 2022).

Given these results, it is evident that the chemical composition of sediments and the presence of thermotolerant coliforms are directly related to land use and management in the studied sub-basins. The observed variations reflect both the natural characteristics of Cerrado soils and the impacts of agricultural activities, highlighting the influence of liming, fertilization, and organic waste contamination on the environmental quality of springs.

Conclusion

The distribution of the analyzed chemical elements varied between sub-basins, indicating influence of land use and occupation. The association between calcium and sub-basin 3 is likely related to liming for sugarcane cultivation, while the significant presence of phosphorus, aluminum, and magnesium in sub-basin 6 indicates fertilizer application. These observations demonstrate the influence of agricultural practices on the environmental quality of springs.

The high values of thermotolerant coliforms in sub-basins 1 and 4, associated with pasture areas, highlight the need for environmental conservation strategies to prevent water resource contamination. Furthermore, the increase in copper, although below legal limits, indicates possible contribution from leaching of agricultural inputs to the analyzed sediments.

Therefore, it is essential to implement sustainable soil management practices and spring conservation measures, with special attention to controlling fecal contamination and mitigating the impacts of agricultural inputs. The recovery of riparian vegetation, preventing cattle access to springs for drinking, and continuous monitoring of sediment quality are fundamental measures to ensure ecological integrity and water availability in the region.

Acknowledgements

FAPEMIG process APQ – 00420-21

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