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CONCENTRATION OF SUSPENDED SOLIDS BY SPECTROPHOTOMETRY

CONCENTRAÇÃO DE SÓLIDOS SUSPENSOS POR ESPECTROFOTOMETRIA

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Abstract

The use of sensors with optical principles designed to estimate sediment transport, and mainly the concentration of suspended solids (CSS) constitutes a robust technique that is currently consolidated and well disseminated in the scientific community, thus configuring an important tool in research, management and control of river basins. The present study, carried out in a water body located near the mouth of the Coxim River - (Coxim , MS), consisted of comparing Nephelometric Techniques and the integrated area of absorbance at wavelengths in the spectrum from 400 to 600 mm (A400-600) and these are measured using a UV/VIS spectrophotometer for indirect determination of CSS. This water body showed characteristics of high sediment input; and for the purpose of possible relationships between the parameters and the characterization of suspended sediments transported by the river, the granulometry (Medium Sand, Fine Sand, Silt and Clay) of the processed samples was determined. The interactions between the parameters to indirectly determine CSS showed the following correlations: ($p<0.001$); Nephelometric with ($R=0.942$) and A400-600 ($R=0.959$); and the particle size fractions, in turn, indicated: (%) of Silt (52.6 ± 17.4), in addition to values close to the distribution of Clay (15.3 ± 6.73), Fine Sand (15.6 ± 10.83) and Medium Sand (16.7 ± 11.84). In this context, according to the results of the residues and correlations, it was observed that A400-600 behaved in a more allusive way compared to the Nephelometric technique in drought and flood events, thus estimating the Detection Limit for CSS (26.47 mg .L^{-1} and 44.67 mg.L^{-1}) respectively.

Resumo

O emprego de sensores com princípios ópticos destinados a estimar o transporte de sedimentos, e principalmente a concentração de sólidos suspensos (CSS) constitui uma técnica robusta que atualmente encontra-se consolidada e bem difundida no meio científico, configurando assim uma ferramenta importante na pesquisa, gestão e controle de bacias hidrográficas. O presente estudo realizado em um corpo hídrico presente nas proximidades da foz do Rio Coxim - (Coxim, MS), consistiu em comparar as Técnicas Nefelométricas e a área integrada da absorbância em comprimento de onda no espectro de 400 a 600 mm (A400-600) sendo estas medidas por meio de um espectrofotômetro UV/VIS para determinação indireta de CSS. Este corpo hídrico apontou características de alto aporte de sedimentos; e para efeito de possíveis relações entre os parâmetros e a caracterização dos sedimentos suspensos transportados pelo rio, foi determinada a granulometria (Areia Media, Areia Fina, Silte e Argila) das amostras processadas. As interações ocorridas dos parâmetros para determinar indiretamente CSS apresentaram as correlações: ($p<0,001$); Nefelométrica com ($R=0,942$) e A400-600 ($R=0,959$); e as frações granulométricas, por sua vez, apontaram: (%) de Silte (52,6 ± 17,4), além dos valores próximos de distribuição de Argila (15,3 ± 6,73), Areia Fina (15,6 ± 10,83) e Areia Média (16,7 ± 11,84). Nesse contexto, de acordo com os resultados dos resíduos e as correlações, observou-se que A400-600 se comportou de modo mais alusivo em comparação a técnica Nefelométrica nos eventos de seca e cheia, estimando desta forma o Limite de Detecção para CSS (26,47 mg.L^{-1} e 44,67 mg.L^{-1}) respectivamente.

1. INTRODUCTION

According to Carvalho (2000), the Concentration of Suspended Sediments (CSS) is characterized by the sediments present in the turbulent flow, which remain in upward and downward movements in the water column , and represent 99% of the total solid load transported by the body. water.

In this process, through the deposition of sediments, significant changes occur in relation to the morphology of water bodies, and in effect, the deterioration of water quality becomes a fact (Carvalho, 1994).

In addition to these, there are also other effects in relation to the transported sediments, Oliveira & Calheiros (2006) mention that many of these sediments are deposited in the periphton region , and depending on the magnitude of the flood pulses and the input of loaded material, they lead to disruption and destruction. ecological imbalance of communities of aquatic microorganisms, thus distorting the environment from both a physical and chemical point of view.

In this sense, the quantitative determination of sediment transport in water bodies constitutes a powerful tool capable of supporting the management and control of river basins, thus providing diagnoses and assistance in monitoring the influences resulting from anthropogenic and/or natural activities, which are responsible for the increase in the amount of sediment transport, mainly erosion.

Currently many techniques for CSS estimation are employed; among which the following stand out: gravimetric techniques, acoustic reflection, laser diffraction, pressure difference, remote sensing response, and finally the optical ones that are widely mentioned in the specific literature (Anderson et al., 2009 ; Sari et al ., 2015).

Among these techniques, the main one that uses optical principles is Turbidity; This is undoubtedly a conventional parameter, widely used for water quality monitoring purposes, and through its interaction with sediments, duly respecting the equipment calibration conditions (Hudson, 2001; Sari et al., 2015) , are strongly correlated, a condition that makes this parameter an important tool for indirectly determining CSS.

water column at an angle of 180°, called Absorbance (cm-1), or, the energy reflected (dispersed) by the body, generally at angles of 45° to 90°, called Nephelometry (NTU) (Anderson, 2005; EPA, 1993, APHA, 2012).

However, for this technique, some recommendations must be considered: the quantity of particles (Kineke and Sternberg, 1992; Sadar , 2005), particle size (Sari et al., 2017), composition (Asano and Sato, 1980; Hillel , 1982; Bunt et al., 1999), color (Sutherland et al., 2000), specifications regarding the sensor's detection angle (Downing , 2006; Anderson et al., 2010) and presence of gases, bubbles and turbulence (Lewis and Eads , 2009, Minella et al., 2008), and depending on the conditions of the sampled medium, the regressions between Turbidity and CSS obtained can be linear, polynomial, multiple and combined.

In several works, initial equations with R2 = (0.55 to 0.98) were obtained, such as the works of Riley (1998) and Minella et al. (2008) respectively.

Regarding the wavelength used in Turbidimeters , it is noted that this parameter is variable, however, most research uses sensors that employ wavelengths in the near-infrared region in the range of 780 to 900 nm, as can be seen in the works by Sari et al. (2017); Harrington and Harrington (2013); Ziegler et al. (2014); Rasmussen et al. (2011). Other sensors also use wavelengths in the Visible region – 400–600 mm – (Pinheiro et al. 2013).

This Visible method is described in the Standard (EPA, 1993), which describes a measurement system with a polychromatic source, based on tungsten light sources, and wavelengths between 400 and 600 nm .

Currently some equipment is equipped with LED light sources; Submersible sensors available on the market that operate in scanning mode in the range (200~750nm) and those equipped with LED lamps, and their combinations between wavelengths allow increasing the range of relationships with other parameters (COD, TOC, COT, NO3 and others), thus improving the accuracy and efficiency of the technique for determining CSS values (Langergraber et al., 2004; Maribas et al., 2008; Brito et al., 2014).

In view of the context exposed above and, bearing in mind the relevance of CSS measurement, this work proposes an alternative using the integration calculation aiming to employ the integrated area between the wavelengths 400 to 600 nm, in intervals 1.0 nm (A400-600).

2. METHODOLOGY

2.1 Study Area

The present study was carried out near the mouth of the Coxim River, an important tributary of the Taquari River, located in the city of Coxim-MS, at a collection point (P), at geographic coordinates 18º32'02" S and 54º44'09 "W (Figure 1).

2.1 Analysis

The samples thus obtained were sent to the laboratory and processed according to the usual methodology (APHA, 2012). CSS (2540.B) was determined in triplicates, based on filtration through a fiberglass membrane washed with ultrapure water, previously weighed after calcination at 550 to 600 °C.

Spectrophotometric scanning (HACH DR6000) was immediately applied to the filtered samples, at a wavelength between 400 and 600 nm , and expressed in integration of the area formed, in unit intervals of 1.0 nm , in a cuvette made of quartz, optical path (1.0 cm), new and unique used during the research. These wavelengths were chosen, as they are used in the Turbidimeter under study.

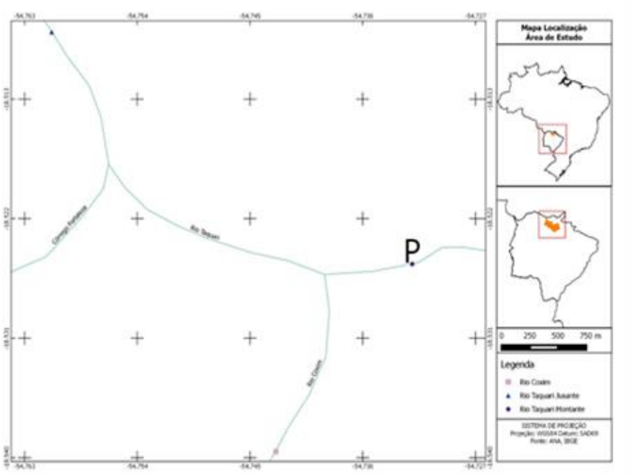

Figure 1. Collection and Monitoring Point (P) - Municipality of Coxim-MS (ANA-IBGE, 2016) modified by the author.

Turbidity values (NTU) were measured using Orion AQ4500 equipment, wavelengths 400 and 600 nm . (In order to calibrate correctly before use with standards 0 to 1000 NTU of Formazine ($C_{17}H_{13}N_5O_3$) supplied by a credited and certified company within the expiration date.

For the purpose of composition (%) of the sediment particle size of the water samples, granulometric characterization was used using the Laser Scattering technique, with a detection range of 0.02 to 2000 μm using the MASTERSIZER 2000 equipment. Water samples were added to a 900 ml Becker, with distilled water core, without the application of ultrasound, and maximum obscuration of up to 20% (Malvern , 2016).

3. RESULTS AND DISCUSSION

According to the classification of Koppen and Geiger (1928), the city of Coxim-MS has an average temperature of 25.5 °C and an average annual rainfall of approximately 1480 mm. The climate has tropical characteristics, presenting seasons with humid summers and predominantly dry winters (INMET, 2017).

During the campaigns, the average monthly accumulated rainfall was 12.95 mm, with a daily maximum of 73 mm and an average ambient temperature (°C) of 26, maximum of 31 and minimum of 20 (INMET, 2017).

There was no rainfall during the collections, but the collection points in the transversal section were variable, due to the constant migration of the river banks, which has characteristics of high sediment supply and a mobile bottom, making it impossible to measure and measure the heights of the rulers. . According to Brito et al. (2013), Teles et al., (2022), the constant migrations of river banks tend to present high amounts of sediment, making it impossible to measure the heights of the rulers, and this type of sediment accumulation is common in some rivers in the country.

To characterize and verify possible interactions with other variables (Turbidity and A400-600), the granulometric composition was classified, according to the Standard (ASTM, 2003), in measurements related to Clay, Silt, Fine Sand and Medium Sand.

The particle distributions (%), for all samples presented the following distributions: Clay (15.3 ± 6.73), Fine Sand (15.6 ± 10.83) and Medium Sand (16.7 ± 11.84), despite presenting close values, apparent deviations occurred when comparing the extracts.

Silt presented the highest percentage (52.6 ± 17.4). These data are in line with those obtained by Teles et al., (2022) who analyzed the water quality of the Mogi-Guaçu river in the state of Paraná, realizing that pH, Total Dissolved Solids (TSD) and Turbidity presented similar deviations for the amount of clay, fine and medium sand in the samples, indicating an increase in turbidity and total solids as the river presented no vegetation cover. Griego et al. (2017), mention that the increase in conductivity is related to the accumulation of polluting loads, turbidity and accumulation of ions and dissolved solids. According to the same authors, the average turbidity values showed a statistically significant difference ($p < 0.05$) between the points collected in the Tanquinho stream in the municipality of Ribeirão Preto/SP, and the increase in turbidity may be associated with the amount of water released. raw sewage and agricultural activities in the region.

CSS concentrations recorded values (220 ± 134.71) $mg.L^{-1}$, reaching the highest value of 528 $mg.L^{-1}$ in (C11F) precipitation season, and for Dry conditions, recorded the lowest value 38 .7 $mg.L^{-1}$ C4F). The A400-600 values were integrated using the linear functions found in the scan ($R>0.98$ and $p<0.001$). To verify the existing relationships between the variables and provide some indirect estimate of CSS, the correlations found between Turbidity and A400-600 with particle size composition and CSS are in accordance with Table 1.

Table 1. Correlation between the variables: A400-600 and Turbidity (NTU) with the particle size fractions and CSS (mg. L-1).

Table 1. Correlation between the variables: $A^{400-600}$ and Turbidity (NTU) with the particle size fractions and CSS (mg. L^{-1}).

	Clay	Silt	Thin sand	Medium Sand	CSS
A400-600	0.633	0.682	-0.762	-0.681	0.959
Turbidity	0.642	0.71	-0.776	-0.706	0.942

Pearson Correlation ($p<0.001$)

The interactions that occurred demonstrate that the Fine and Medium Sand class correlates negatively with Fine Sand and Turbidity, not influencing the signal response as highlighted by Conner and Visser (1992), unlike Clay and Silt. The works of Gomes (2013), Merten et al. (2014), Sari et al. (2017), demonstrated that finer fractions of particles (Silt and Clay) influence the Turbidity reading, corroborating the values listed here with correlation. Regarding the estimation of CSS in function of the Techniques employing optical sensors, excellent correlations were verified ($p<0.001$), for Turbidity R2= 0.89 and, with a better result for A400~600 R2=0.92, in line with the results obtained by Gomes (2013).

In Figures 2 and 3 are the regressions, prediction for Turbidity and A400~600, respectively.

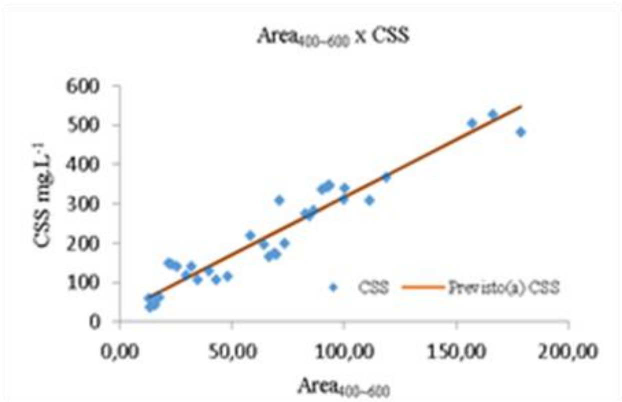

Figure 2 . Regression plot (A400~600) nm x CSS.

The Root Mean Square Error (RMSE) for the spectrophotometric variable was lower than the nephelometric one (37.52 and 44.73) respectively.

The CSS detection limit for the variable Turbidity and A400-600 starts above (44.67 and 24.77) mg. L-1 respectively, although the values found present higher concentrations, not being recommended for measurements in eventual waters with smaller amounts of sediments. These results are in line with those obtained by Silva et al., (2021) who, in the detection of CSS for the variable turbidity and A400-600, highlight that measurements with smaller amounts of sediment made the analysis results difficult.

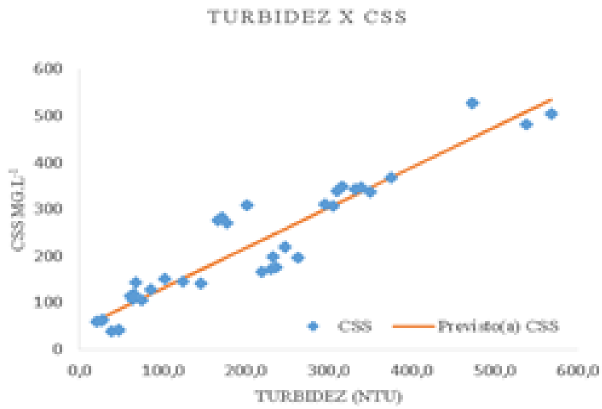

Figure 3. Regression graph (A400~600) nm x CSS

The CSS response behavior, due to the 90 and 180 degree detection angle of the equipment used, obtained similar responses, obtaining a simple linear regression prediction ($F<0.05$). Sari et al. (2015) reports in their work that the characteristics of particles in relation to size, for example, have the same interaction as the response of sensors in different equipment, with small variations depending on the detection angle and wavelength used, contrary to what was reported by Anderson et al. (2010), in which all equipment characteristics, emission angle, spectrum detection, and even calibration, are unique, generating significant values.

According to the authors Medeiros et al., (2016), Martins (2022) highlighting that for the physical-chemical analysis of heavy metals, total solids, turbidity and conductivity, standardization and calibration of the wavelength influence the results, being Care must be taken when analyzing, collecting and equalizing the device.

Turbidimeter Sensor , which has a detection angle of 90°, has greater sensitivity to sediment response in a variable range of sediment particle sizes, which was found in this work, which corroborates the assumption of Merten et al. (2014). The same author describes that sensors at 180° obtained less interference with sensors positioned at 180° to the light emitter, an angle used by A400~600.

As for scanning the spectrum, the method presented satisfactory results in accordance with the above, with adequate correlation and residues, becoming an optional tool for determining CSS. Some sensors that use the same optical signature technique in a larger spectral range, between 200 and 750 nm (Brito et al. 2013; Maribas et al., 2008; Langergraber et al. 2004), have consolidated the method for online monitoring purposes. line .

For Langergraber et al. (2004), the spread of the dispersion promoted by the type of sediment depends on the wavelength, thus, as the absorbance collection range for A400-600 is greater, it would somewhat attenuate possible non-reproducible deviations.

3 FINAL CONSIDERATIONS

According to the data obtained, it can be concluded that spectrophotometric techniques can be a viable alternative for determining Suspended Solids in water bodies.

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