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Water-dispersible colloids extraction methods: combining methodological data analysis and batch experiments to explore trends, feasibility, and impact on colloidal characteristics

Jay Carl A. Cacerez[1*], Antonio De Matteis[1], Rafaella Chiarella[1], Jens Kruse[1], Anne E. Berns[1], Lutz Weihermüller[1] and Nina Siebers[1]

Abstract

Background Water-dispersible soil colloids (WDC) play a significant role in various soil processes, such as in nutrient storage and release. Therefore, it is essential to investigate and characterize WDC to understand their behavior and characteristics. However, a diversity of methods used to extract WDC for characterization is found in literature, which consequently makes interstudy comparison of colloidal data challenging. In this study, we analyzed methodological data on WDC extraction obtained from literature through Principal Component Analysis (PCA) and K-means clustering to examine methodological trends and similarities. Selected extraction methods from the methodological clusters (and the type of dispersant used) were evaluated experimentally to assess how different extractions influence WDC characteristics, and in terms of their feasibility.

Results PCA of methodological information showed that most methodological parameters were drivers of extraction method diversity. From the K-means clusters of the four Principal Components, three extraction methods were identified for comparison. The first method employed sedimentation and centrifugation to separate the WDC fraction, yielding 0.2–0.4% (w/w) of WDC relative to the fresh soil mass. The second method only used sedimentation and extracted the highest WDC quantity (0.6–2.0%), but with the highest proportion of particles with diameters > 1000 nm. The third method involved centrifugation and filtration, extracting 0.02–0.08% of WDC, with an average maximum colloidal particle diameter of 638 nm. While the use of different dispersants did not have an influence on WDC yield, it influenced the particle size distribution (PSD) of WDC extracted, specifically in organic soil. Furthermore, the individual influence of the extraction method and dispersant and their interaction effects on WDC elemental composition vary with soil and the element of concern.

Conclusion Our findings demonstrate that extraction methods influence WDC characteristics in terms of yield, PSD, and elemental composition. The type of dispersant also affects the PSD of WDC in organic soil, and its influence on elemental composition varies depending on soil type and elemental component. Among the extraction methods compared, the first method emerged as the most balanced and reliable approach in extracting WDC in terms of yield and feasibility.

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*Correspondence:
Jay Carl A. Cacerez
j.cacerez@fz-juelich.de

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Introduction

Soil colloids refer to soil particles with diameters between 1 and 1000 nm and are typically composed of clay minerals, Fe and Al (hydr)oxides, and organic substances [1]. Due to their high surface area and reactivity, they significantly influence the soil physicochemical properties [2] and several soil processes. Additionally, because of their strong sorption capacity, they influence nutrient release and storage and thus play a significant role in the biogeochemical cycling of nutrients [3, 4]. They also contribute to aggregate formation and stabilization, which is important for good soil structure and overall soil stability [5, 6]. Understanding the release of colloids following aggregate breakdown and their role in nutrient transport in soil is essential, given their environmental and agricultural relevance.

Due to the complex nature of soil systems that stems from their chemical and physical heterogeneity [7], the study of soil colloid formation and transport is particularly challenging. The existing numerical models on colloid transport and dynamics are usually based on experiments that use engineered colloids. In addition, the upper surface of the soil is typically regarded as the colloid source, as colloidal dispersions are introduced at the top of the column in many simulations and experimental studies [8, 9]. Meanwhile, the in situ release of colloids remains the primary source of mobile colloids in soils and groundwater [10], which occurs especially after soil aggregate breakdown by disruptive forces, e.g., precipitation and freeze–thaw events [11, 12]. With the advancement in colloidal research, there is a potential to further develop and refine the existing models to accurately mirror the behavior of natural colloids. A more accurate simulation of colloidal behavior requires a more accurate collection of empirical data to parameterize and validate these models [13].

Water-dispersible colloids (WDC) are particularly relevant in the context of increasing intensity and frequency of precipitation fueled by climate change. These mobile colloids are released following the disintegration of soil aggregates under disruptive forces and can be transported within the soil profile [9, 14]. Because of their dynamic nature and properties, WDC can enhance the transport of nutrients, particularly phosphorus [3], nitrogen [15], and organic carbon [16] in the vadose zone. Thus, WDC can counteract carbon sequestration and disturb nutrient bioavailability in the soil and can enhance nutrient migration into aquatic environments. Determining the quantity and characteristics of WDC is a prerequisite for understanding the nature of soil colloids. However, several methods have been described in literature for extracting WDC in soils, and using different methods may yield different quantities and characteristics of WDC, which may lead to varying interpretations for understanding soil colloids. In this context, a consistent approach in extracting WDC could aid in generating reliable and comparable empirical datasets, thereby increasing confidence in model predictions for colloid genesis and transport. Integrating the outputs generated using standardized extraction methods into transport models has the potential to enhance the simulation of both applied and naturally occurring colloids. Improved simulations can facilitate a more comprehensive understanding of colloid dynamics and transport under near-natural conditions.

WDC is classically investigated using column [9, 17, 18] and batch experiments [14, 19, 20], which are useful in investigating colloidal behavior and interactions in soil and environmental systems. The use of column and/or batch experiments entirely depends on the specific research objectives. Column experiments are commonly used for investigating colloidal transport and leaching processes [9, 17]. Batch extraction experiments, which are the focus of this study, are employed in quantifying colloidal release and offer an accessible and straightforward means to investigate the potential for colloid mobilization [21]. There are also several analytical techniques employed to characterize soil colloids, such as dynamic light scattering (DLS) [18, 22], field-flow fractionation (FFF) [23, 24], and single-particle inductively coupled plasma time-of-flight mass spectrometry (SP-ICP-TOF–MS) [25]. DLS is employed for determining hydrodynamic size, particle size distribution, and colloidal stability [26]. FFF is usually employed in separating and sizing environmental colloids based on their diffusion coefficients [26]. SP-ICP-TOF–MS can analyze elemental ratios of individual colloidal particles [25]. These techniques have advanced our capability in examining and characterizing soil colloids in high resolution and sensitivity, especially in the aspect of their size distribution and composition [25, 27, 28]. However, they are limited in widespread laboratory applications due to their high operational cost or specialized instrumentation requirements [25, 29].

WDC are operationally defined as colloids released from soil aggregates upon re-suspension in water without introducing aggressive mechanical forces (e.g., sonication). It provides an estimate of the colloidal fraction that can be mobilized under natural disruptive events (e.g., precipitation events) [18] and is likely to participate in transport processes. Furthermore, the use of high energy inputs during extraction disrupts soil aggregates and may result in the detachment of colloids that are otherwise occluded in the soil matrix within the soil structure [30, 31]. These occluded colloids have limited mobility in comparison to WDC under natural disruptive conditions. As documented in literature, WDC extraction through batch experiments conventionally involves shaking followed by the separation of colloidal dispersion using sedimentation, centrifugation, filtration, or a combination of these techniques. Existing methodologies in literature still show inconsistencies; there is no consensus on the optimal protocols for extracting WDC nor on the colloidal size range definition. Per International Union of Pure and Applied Chemistry (IUPAC) definition, colloids are particles with diameters ranging from 1 to 1000 nm [32, 33]. Nonetheless, many studies identify soil colloids as particles with a diameter smaller than 2 µm [34, 35]; others lack an explicit size cutoff or distinction between dissolved and colloid fractions [16, 36], or the minimum size threshold also depends on the filter pore size used in the extraction process [37, 38]. These inconsistencies present a critical gap in colloid research and consequently hamper our understanding of colloid-associated nutrient transport and dynamics. Moreover, these variabilities may result in unreliable interpretation and application of soil colloidal data for nutrient and environmental management. Considering the dependability of conventional WDC extraction methods in soil colloid research, it is necessary to improve their reliability and to promote the use of a consistent extraction method. Establishing a widely accepted method helps in improving colloidal data comparability and reproducibility and in understanding the role that colloids play in soil and in broader environmental systems.

Therefore, this study explores the methods used in extracting WDC from soils in literature, whereby a dataset encompassing different experimental conditions reported for extracting WDC in selected published papers were constructed. Through multivariate analysis of the constructed methodological dataset, the underlying methodological trends and similarities/differences and the key experimental parameters influencing the variability in WDC extraction methods in literature were examined. The results of this database analysis formed the basis of the experiments where selected WDC extraction methods were compared in terms of their feasibility and impact on WDC quantity and characteristics. We hypothesized that different extraction methods employing different shaking and colloid separation steps produce different colloidal yields, size distribution, and elemental composition. Moreover, dispersants with different ionic strengths can further modulate these effects by influencing colloidal stability and aggregation behavior. We expect that using an electrolyte solution to represent natural wetting through rainwater infiltration has an influence on the colloidal yield and properties in comparison to using water as a dispersant.

Materials and methods

Literature review and database creation

A comprehensive search for published papers on WDC was conducted, yielding 84 and 77 science-indexed articles from Web of Science and Scopus, respectively. The following search keywords were used: “water-dispersible,” “soil,” and “colloids.” In addition, eight papers were sourced through citations of the found publications. A total of 169 collected publications were screened systematically based on the key criteria shown in Section S1.

The extracted information from the selected papers for the construction of a database included details on sample material, sampling details, soil physicochemical properties, sample preparation, WDC extraction method from soils and sediments, and the quantity of WDC obtained (Table S1). The quantity of WDC extracted was often not measured (or reported), and the obtained colloidal suspensions were typically directly used for colloid characterization [39] or applied to column experiments [40, 41]. In several papers, the WDC quantity was only graphically presented; this information was reconstructed by digitizing the graphs using WebPlotDigitizer v.4.6 [42]. The metric for each methodological variable used to build the dataset was converted to common units for uniformity and comparability. Consequently, some studies were excluded for further data analysis when unit conversion of a necessary methodological variable was impossible.

Laboratory WDC extraction

Based on Principal Component Analysis (PCA) and cluster analysis of methodological data from 27 studies, the most prevalent extraction methods within the identified clusters were experimentally evaluated for their impact on WDC characteristics, including yield, particle size distribution (PSD), and element composition.

Three soil samples, representing a composite of organic-rich (O-horizon), organo-mineral soil (A-horizon), and mineral soils (B-horizon), were used to compare conventional WDC extraction methods in terms of their efficiency in different soil types, specifically in soils with varying organic matter content and texture. The soil samples were collected from the Wüstebach catchment site (50° 30’ N, 6° 20’ E) in Germany, close to the German–Belgian border [43]. The field-fresh soils were sieved to < 2 mm and then stored at 4 °C prior to analysis to preserve their physicochemical characteristics (Table S2). For WDC extraction, 5 g of the prepared soil samples were used. The extraction and experimental tests were performed in triplicate to assess the precision of the extraction methods.

To disentangle the influence of ionic strength on WDC characteristics, two dispersants were used: high-purity water (18.2 MΩ cm, Total $C_{org} < 3$ ng g^{-1}, Merck Millipore, Germany) and electrolyte solution (Section S2). The electrolyte solution was used to represent the ionic composition of natural rainwater during infiltration and wetting events to reflect conditions where WDC could be naturally mobilized. This study focused on colloids with diameters < 1000 nm. The duration of the separation steps, including centrifugation or sedimentation, was calculated using Stokes' law, considering the average bulk densities of the mineral (2.65 g cm^{-3}) and the organic (1.6 g cm^{-3}) materials. The settling times for sedimentation (7.5 cm sedimentation distance) and centrifugation [3000 Relative Centrifugal Force (RCF)] were calculated using Eqs. 1 and 2, respectively (Section S3). The filtration step involved the use of polyethersulfone (PES) syringe filters (J.T. Baker, USA) with a pore size of < 1 µm. Before performing any of these WDC separation steps, the soil suspensions obtained after shaking were evaluated for their volume-based PSD using a particle analyzer (LA-950, Horiba, Kyōto, Japan) [44].

To determine the WDC quantity gravimetrically, an aliquot of the colloidal dispersion obtained after the separation steps was oven-dried at 35 °C and weighed. Since the gravimetric mass of WDC does not provide information on the size of WDC extracted, the DLS technique was used on separate aliquots to determine the colloidal PSD and to assess how the methods accurately extract WDC to the defined cutoff diameter (< 1000 nm). The Malvern ZetaSizer Nano-ZS (Malvern Instruments GmbH, Germany) was used for DLS measurements. Asymmetric Flow Field-Flow Fractionation (AF4, Postnova Analytics, Landsberg, Germany) equipped with 1 kDa (~ 0.66 nm) polyethylenesulfone membrane and 350 µm spacer was used to determine the elemental composition (Table S3). The organic C (C_{org}) content of the colloidal dispersion was measured using the AF4 coupled to an C_{org} detector (OCD; DOC Labor Dr. Huber, Karlsruhe, Germany). In addition, the Al, Si, and Fe contents were measured by AF4 coupled to an inductively coupled plasma–mass spectrometer (ICP-MS; Agilent 7500, Agilent Technologies) [45].

Statistical analysis

All statistical analyses were conducted using R version 4.4.0. PCA was employed to analyze and select the relevant experimental variables for WDC extraction in soils from the constructed database. The *prcomp*-function from the base R *stats* package was used to perform PCA on the complete data on methodological parameters and sample information. The categorical variables were converted into quantitative variables for PCA using dummy encoding, transforming categorical variables with *n* categories into *n* new binary variables. In this study, *n*-1 new binary variables were used to reduce multicollinearity. In the next step, the data were standardized (mean = 0; standard deviation = 1), ensuring that the variables were on the same scale and the data were amenable to PCA. The important principal components (PC) for further analysis were selected based on the scree plot and cumulative variance criteria. The factor loadings of the variables from the selected PC were analyzed to identify the most relevant experimental variables contributing to the variability in the methods for extracting WDC. The K-means algorithm was applied to the scores corresponding to the first four PCs using the *kmeans*-function for clustering, with the optimal number of clusters (k) determined using the gap statistics metric. From the identified K-means clusters, medoid observations were determined to identify representative WDC extraction methods for experimental comparison. In addition, the prevalent methods in each cluster were identified to aid in the selection of extraction methods for comparison.

For the statistical analysis of experimental data, a significance level of $p = 0.05$ was used. The experimental data for each soil sample were tested for normality of distribution using the Shapiro–Wilk test (*shapiro.test*-function) and for homogeneity of variance using Levene's test (*leveneTest*-function from *car* package). If the assumptions of normality and equality of variance were fulfilled, a two-way Analysis of Variance (ANOVA) was employed using the *aov*-function in the base *stats* package to evaluate the effects of the extraction methods and dispersants on WDC characteristics. Post-hoc comparisons with Bonferroni-Holm correction were further performed if significant differences occurred in each factor. A pairwise t-test was performed using the *pairwise.t.test*-function to examine the effect of methods in a dispersant and the effect of dispersants in a method. If the assumptions were not met (i.e., normal distribution), Aligned Ranks Transformation ANOVA (ART) was performed using the *art*-function in the *ARTool* package.

Identification of the best WDC extraction method

The feasibility of the selected extraction methods from cluster analysis was evaluated based on cost, time, effectiveness, and precision, with each criterion scored on a scale of 0 to 10, where 10 represents the best performance. The score for the cost was based on whether the method used essential equipment or materials for WDC extraction (i.e., shakers, centrifuge, filters). Time was based on the duration required to process a batch of samples for each method. Effectiveness was assessed by the quality of WDC size distribution, considering the cumulative intensity of particles within and outside the < 1000-nm fraction and the maximum size extracted within the colloidal fraction. Precision was determined using the coefficient of variation (CV) of the WDC quantity extracted by each method. The equations used for the calculation of feasibility criteria scores are provided in Section S4.

Results and discussion

Methodological database analysis

Out of 169 papers gathered, only 36 studies matched the strict criteria defined for selecting appropriate studies. This study focused primarily on investigating experimental parameters related to WDC extraction. From the 36 papers selected after screening the collected papers, only the methodological data extracted from 27 papers were used for further analysis. The nine papers excluded lacked sufficient detail, particularly regarding the extraction procedure for WDC. In many cases, the absence of critical details, e.g., shaking duration, centrifugation intensity, rendered these studies unsuitable for inclusion. For example, data on the relative centrifugation force (RCF) is needed for the database creation, but details on the revolutions per minute (RPM) or the type of centrifuge used are missing, which limits unit standardization in the database. This lack of comprehensive reporting highlights the importance of consistently documenting and reporting relevant experimental information to facilitate reliable comparisons.

The selected studies generally focus on the colloidal properties, behavior, and role of colloids in soil processes and were published in 17 scientific journals between 1996 and 2023. During these years, the number of publications involving the extraction of WDC has steadily increased. This increase reflects the technological advancements in characterizing colloids and the growing need to understand colloidal systems. Regionally, the selected studies originated from 11 different countries, with most coming from temperate climates. Studies from Germany ($n = 9$), the USA ($n = 5$), and China ($n = 3$) dominate. From the 27 studies, a total of 317 data entries or observations were obtained, comprising a complete dataset on 14 variables of soil characteristics and experimental information. This data was used in the later step for PCA.

The soils from the studies with available data had pH values ranging from 3.3 to 8.3 and C_{org} content ranging from 0.0 to 13.6%. The clay, silt, and sand content ranged from 1.0 to 75.0%, 1.5 to 78.2%, and 2.0 to 97.2%, respectively. From the total data entries, 9% were sampled from the litter/humus layer, 8% from the topsoil, 15% from the subsoil, and the remainder were either sampled from the parent material, sediment, or no data was reported.

From the selected studies, 23 distinct WDC extraction methods were identified, each varying in experimental parameters (Table S4). In some cases, unique extraction methods were derived from the same study as different levels of procedural parameters were used and tested. These variations were treated as separate methods to reflect their distinct methodological conditions. From the data gathered, the soil-to-dispersant ratios ranged from 1:20 to 1:2 (w/v) while shaking durations varied from 5 min to 24 h. The shaking intensities used in the selected studies spanned from 10 to 200 rotations per minute (rpm). Overhead and horizontal shakers were among the shaker types used. A range of separation techniques, including sedimentation, centrifugation, and filtration, was either applied sequentially or in combination to isolate the desired colloidal fraction from the soil suspension. These methodological differences, including variations in sample preparation (i.e., storage and drying), shaking times, and separation steps (i.e., filtration and centrifugation), foil interstudy comparison of colloidal data. Such inconsistencies can directly influence the quantity and quality of WDC extracted [20, 46].

Typically, PCA is applied to experimental results and rarely used on methodological data. In this study, PCA was used to visualize underlying trends in the conventional methods for extracting WDC from soil or sediment. Its application can help in examining methodological diversity and grouping similar methodologies across scientific studies. To our knowledge, PCA has not been utilized before in this context, particularly in colloidal studies. However, the limited procedural data for WDC extraction obtained in this study may result in misrepresentation of methodological diversity in literature. Nevertheless, PCA can still be used as an exploratory tool to identify methodological trends, such as the correlations among the existing extraction methods. Performing PCA accentuated the need for complete reporting of necessary methodological information for better interstudy data comparisons.

The number of resulting Principal Components (PC) considered for identifying the relevant variables was determined based on their Eigenvalues and the cumulative variance they explained. Eigenvalues represent the amount of variance captured by each PC, and components with Eigenvalues > 1 were retained for further interpretation, following Kaiser's rule [47] (Fig. S1a). The retained PCs accounted for more variance than any of the original variables in the standardized data set. The first, second, third, and fourth PCs accounted for 38.2%, 19.0%, 14.0%, and 10.6% of the total variance, respectively, cumulatively explaining 81.7% of the variance (Fig. S1b).

The experimental parameters reported in the studies driving the heterogeneity in WDC extraction methods include sample type, sample drying, dispersant type, sample-to-dispersant ratio, shaking time, shaking intensity, suspension dilution after shaking, and the separation steps (Table 1). These variables exhibited high absolute loadings (> |0.7|), indicating that their variabilities are well-represented by the retained PCs, and that they highly influence the variability in the methodologies used for WDC extraction. In the first and third PCs, the variables with the highest predictive power were the extraction variables (shaking time, shaking intensity, sample-to-dispersant ratio, dispersant type, and suspension dilution) and the separation variables (sedimentation, centrifugation, and filtration). Sample preparation, particularly sample drying, was the most important variable in the fourth PC. The second PC highlights the importance of sample type and composition, indicating that inherent soil properties are a critical factor in driving methodological differences. The variability in sample types in the selected studies reflects different physical and chemical properties of the samples, which could influence their behavior during the extraction and separation steps. Additionally, these samples were also collected from various locations with varying land uses, vegetation, and climatic conditions. The extraction methods were often tailored to the specific characteristics of the samples, the goals of the studies, and the equipment availability in the respective laboratories.

Analysis of the first two PCs revealed an inverse relationship between shaking time and both the sample-to-dispersant ratio and shaking intensity (Fig. 1). This result suggests that studies employing higher sample-to-dispersant ratios tended to use higher shaking intensities but shorter shaking times. Additionally, higher sample-to-dispersant ratios were often associated with the application of a sedimentation step and the absence of a filtration step. They also tended to dilute the suspension after shaking and before colloid separation. With sample drying, studies typically employed air-drying for mineral soils and oven-drying for organic samples. Moreover, studies that used water as the dispersant during shaking were more likely to include a centrifugation step.

Samples from studies applying the same extraction protocol were represented as converged points in the PCA subspace for individuals or observations. This observation suggests that studies with similar methods for extracting WDC tend to exhibit comparable

Table 1 Loadings of the first four Principal Components (PC) obtained for the methodological variables taken from the literature database

Methodological variable	PC1	PC2	PC3	PC4
Sample				
Mineral soil	−0.319	**0.813**	0.128	0.192
Organic layer	0.417	**−0.750**	−0.080	−0.318
Sample drying				
Undried	0.421	−0.252	−0.140	**0.843**
Air-dried	−0.528	0.586	0.145	−0.526
Oven-dried	0.300	−0.634	−0.031	−0.511
Dispersant	0.144	−0.124	**0.883**	0.118
Sample-to-dispersant ratio	**0.851**	0.373	0.127	0.019
Shaking time	**−0.851**	−0.299	−0.022	0.131
Shaking intensity	**0.841**	0.139	0.082	−0.050
Dilution step after shaking	**0.936**	0.221	0.130	−0.088
Sedimentation step	**0.898**	0.232	−0.197	−0.063
Centrifugation step	0.159	−0.013	**0.894**	−0.075
Filtration step	**−0.800**	−0.138	0.255	0.027
WDC size (max.)	−0.033	0.484	−0.404	−0.219

Values in bold represent high absolute loadings (> |0.7|)

The four PCs were obtained through principal component analysis of the methodological dataset

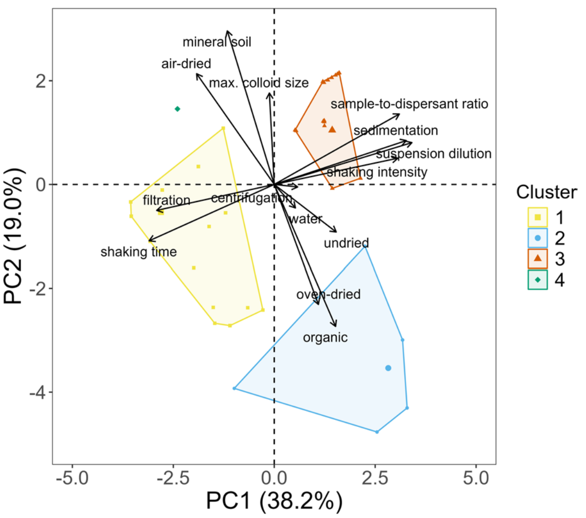

Fig. 1 Factor plot of PC1 versus PC2 showing the relationships between methodological variables taken from the literature database and the groups of observations identified using K-means clustering. Cluster sizes are as follows: Cluster 1 = 9 studies; Cluster 2 = 4 studies; Cluster 3 = 14 studies; Cluster 4 = 1 study. Note: The sum of studies across clusters does not equal the number of studies evaluated for Principal Component Analysis and cluster analysis, as some studies employed multiple methods and thus may appear in another cluster

experimental parameters, as reflected in their proximity within the PCA subspace, and hence can be clustered. Using K-means clustering, four clusters were identified (Fig. S2). Four WDC extraction methods were identified from K-means clusters one to four based on medoid determination, which were designated as Methods C, A, T, and B, respectively (Table S5). Additionally, the methods evaluated were selected based on how many studies employed them in each cluster. Both Methods T and A are similar except for their shaking times, which are 390 and 360 min, respectively (Table S4). Considering that there is only a 30-min difference between the shaking times, and Method A is the most prevalent in clusters two and three, Method A was used to represent these two clusters. In the end, Methods A, B, and C were used for experimental comparison out of the 23 extraction methods. These three extraction methods had the highest number of studies employing them for WDC extraction (Fig. 2a). Each method represents a distinct combination of extraction and separation parameters (Fig. 2b). The identified methods adequately represent the diversity of methods used for WDC extraction in soils, making them ideal for comparison in terms of their impact on soil colloid characteristics.

Method A was the most versatile among the three, as it had been applied to mineral soils collected from areas with diverse land uses and climates and a wide range of soil properties, including acidic to basic pH and high to low clay content. Method B and C were used exclusively for mineral soil samples, whereas Method A was also employed for organic soils. Method C was characterized by the longest shaking time among the three, but it had the lowest shaking intensity and was the only method that included a filtration step. In contrast, Method A used the highest shaking intensity and the shortest shaking time and included a suspension dilution step after shaking. These observed characteristics aligned with the trends identified in the PCA correlation plot, further validating the relationships between shaking parameters and separation techniques.

Experimental section: comparing identified extraction methods for WDC characteristics

Shaking equipment and suspension particle size distribution

The three extraction methods identified by PCA and K-means clustering differed in their experimental parameters, such as the type of shaker used. The impact of shaking on the characteristics of WDC extracted may be influenced by the type of shaker used due to the differences in the shaking mechanism and motion patterns, especially at lower shaking intensities. Methods B and C typically use reciprocal shakers, a type of horizontal shaker that shakes the samples in a linear, back-and-forth motion. However, the horizontal shaker (SM-30 Control, Edmund Bühler GmbH, Germany) available in our laboratory could not replicate the shaking intensities required for Methods B and C, which are 29 and 10 rpm, respectively. Therefore, an overhead shaker (Multi Bio RS-24, Biosan SIA, Latvia) was used for these two methods, which rotates the samples around a fixed axis. The

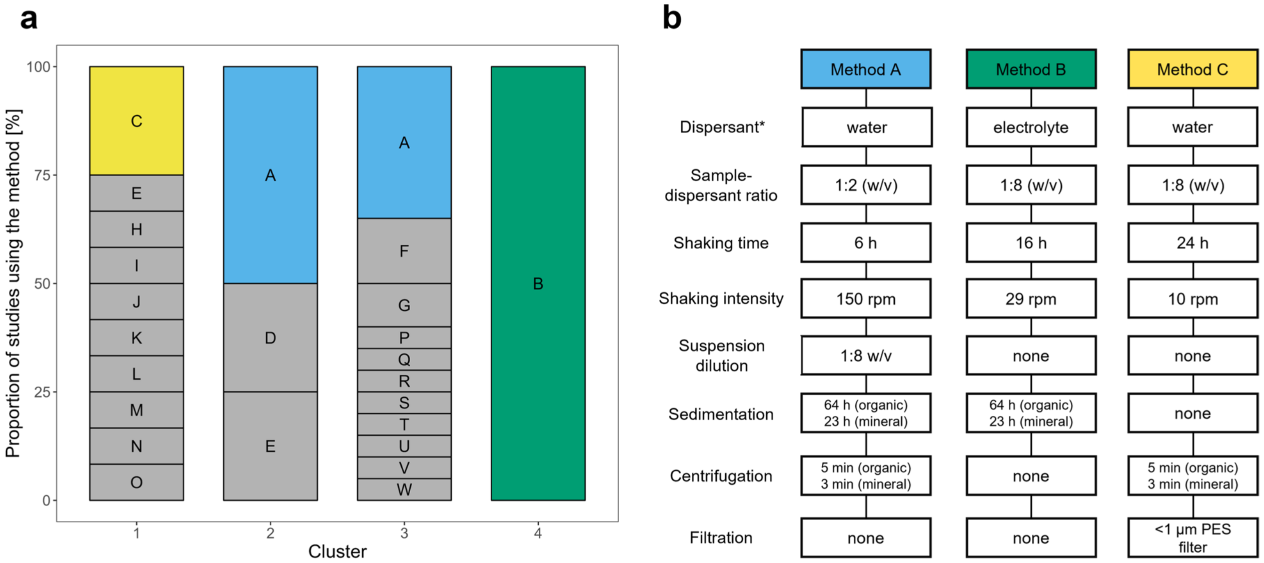

Fig. 2 **a** Proportion of studies employing the identified conventional water-dispersible colloids (WDC) extraction methods within each K-means cluster of Principal Component Analysis observations. **b** Experimental details of the three identified extraction methods to obtain < 1000 nm WDC from soil suspension. *Both water and electrolyte dispersants were used for the comparison of the three extraction methods

use of an overhead shaker for both methods was advantageous, as it ensured resuspension of particles during shaking through gravitational inversion. Using a horizontal shaker at these low shaking intensities can lead to reduced shear forces and to limited particle-dispersant contact due to settling of heavier particles at the bottom. For Method A, which required higher shaking intensity (150 rpm), horizontal shakers were used consistently in the collected studies. Horizontal shakers move samples in a horizontal plane, either in orbital or reciprocal motion. The higher shaking intensity used in this method ensured that the denser particles remained suspended and interacted with the dispersant, promoting colloid extraction while using a horizontal shaker.

The influence of the differences in the shaking parameters (soil-to-dispersant ratio, rpm, duration, and type of shaker) of the three methods on the three soil samples was assessed by analyzing the PSD of the soil suspension obtained after shaking. The PSD of the suspensions was predominantly in the microaggregate

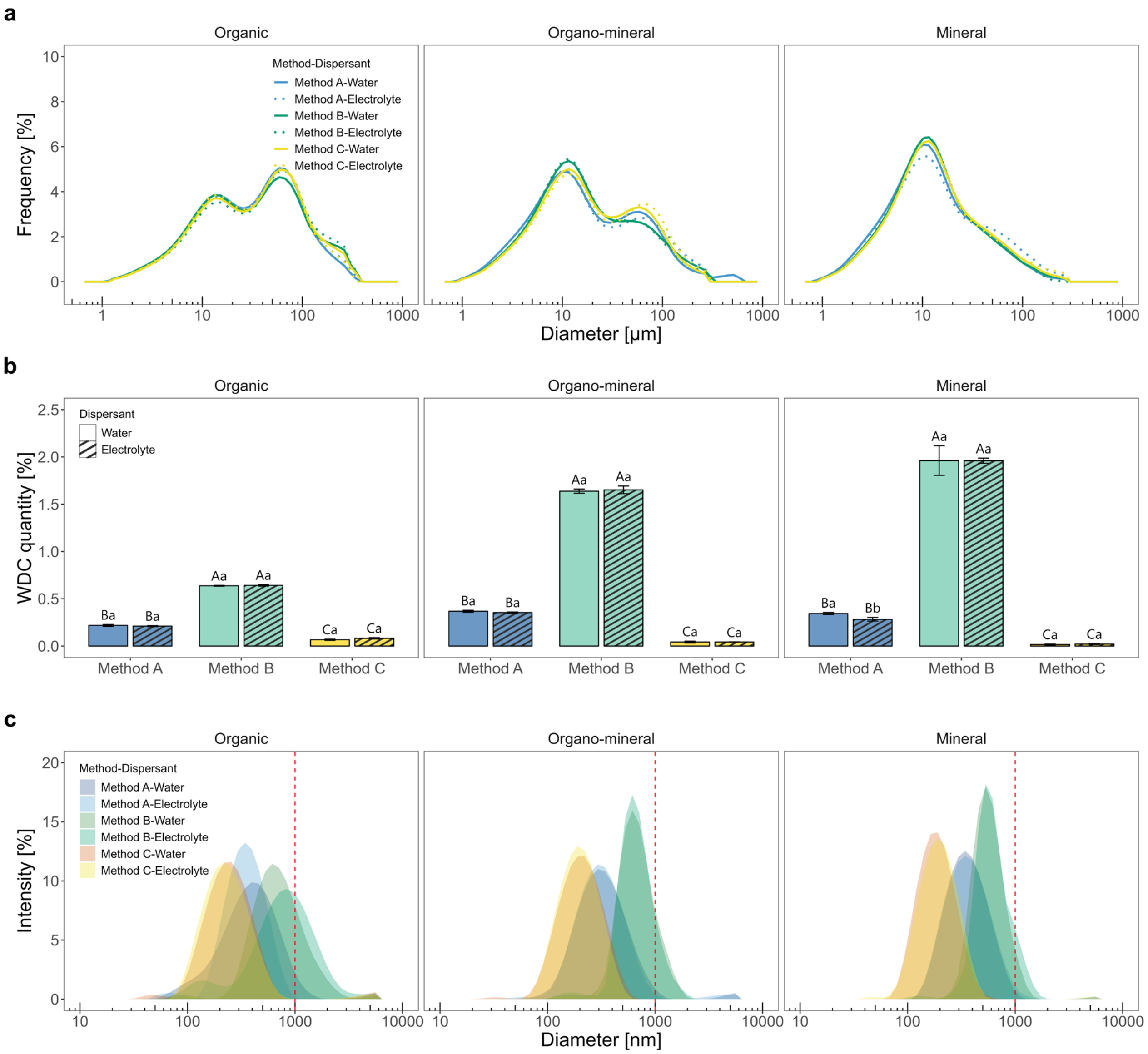

Fig. 3 **a** Particle size distribution of the soil suspensions obtained after shaking of organic, organo-mineral, and mineral soil samples, **b** quantity of extracted water-dispersible colloids (WDC). Capital letters in the plot indicate comparison of using different methods within the same dispersant; small letters indicate comparison between dispersants within the same method; different letters indicate statistical difference at $p = 0.05$ using pairwise t-test, and **c** colloidal particle size distribution obtained after the complete WDC extraction process using the three identified WDC extraction methods (Methods A, B, & C) and two dispersants (water & electrolyte)

size range (< 250 µm). The median particle size of the obtained suspensions did not vary greatly among the three extraction methods (Fig. 3a). This finding indicates that the differences in the shaking parameters among the three methods did not result in substantial differences in PSD. The disruption of the architecture of larger soil aggregates to form smaller constituents may have been similar for the three methods, resulting in similar PSD. In general, microaggregates are a heterogeneous assemblage of materials of mineral, organic, and biotic origin that are bound together, which consequently makes them resistant to intense mechanical and physicochemical stresses [48]. Despite the long shaking time of Method C, which can result in more fragmentation of soil aggregates [49], it also had the lowest shaking intensity. Method A had the shortest shaking time; however, it had the narrowest sample-to-dispersant ratio and the highest shaking intensity (Fig. 2b), which may have enhanced aggregate destruction even at the shortest shaking time. Such conditions of higher suspension concentration and more intense shaking might result in higher shattering and abrasive actions among soil particles and aggregates [50]. While mechanical shaking was used to facilitate colloid dispersion and represents a moderate form of agitation to disrupt aggregates, it is important to note that elevated shaking intensities may overcome the attractive forces that bind particles within aggregates. The increased hydrodynamic shear and collision energy from intensive shaking can result in the mobilization of colloids that would otherwise remain attached to the soil matrix under natural disruptive conditions (e.g., precipitation events), leading to the overestimation of WDC extracted.

Differences in the PSD of the soil suspensions were mainly observed between soil samples (Fig. 3a). Previous work has shown that superficial horizons often contain a mix of particle sizes due to organic matter accumulation and biological activity [51] and higher exposure to mechanical weathering. Consistent with this, our experimental results have indicated that soils from these layers exhibited broader particle sizes. For organic soil, which was obtained from Oh-horizon, a bimodal PSD was observed. The first peak, with smaller diameters, likely corresponds to stable mineral components, while the second peak, with larger diameters and in higher proportion, represents aggregated organic components, as previous studies have shown that organic matter compounds act as gluing agents and promote aggregation [48, 52]. A similar bimodal distribution was observed in the organo-mineral soil, which was from Ah-horizon, though with a higher proportion of mineral components than the organic components. Conversely, the mineral soil displayed a unimodal distribution dominated by mineral particles characterized by smaller diameters. The mineral soil was obtained from B-horizon, which usually exhibits more uniform distributions in particle sizes. This observation is in line with previous reports that B-horizons usually exhibit more uniform particle size distributions due to the illuviation of finer particles from upper horizons [53]. Overall, the variation in the particle size distributions of the suspensions obtained after shaking the three samples reflects the diversity of the soil samples used in the study—organic, organo-mineral, and mineral soils.

Quantity of extracted WDC

In terms of the quantity of extracted WDC (Fig. 3b), Method C yielded the lowest amount (0.02–0.08% fresh weight basis) across all samples, likely due to colloid loss during filtration (Method A and B did not filter the suspension). Filtration may underestimate WDC quantity extracted as colloidal particles can be retained on the filter due to the formation of filter cakes [54, 55]. Colloids can be irreversibly adsorbed to the filter membrane or coagulate at the filter surface, trapping smaller particles within the coagulated matrix [10]. Filtration can also remove more material from soil suspensions than centrifugation when processing mineral and organic-rich soil samples [56]. Although a centrifugation step was used beforehand in Method C to reduce coarse particle deposition on the filter and allow more colloidal particles to pass through [57], this approach still resulted in the lowest WDC quantity. Therefore, the results obtained with Method C suggest that a higher mass loss of WDC occurs compared to other methods, highlighting the insufficiency of centrifugation as a pretreatment to mitigate WDC loss during filtration.

As mentioned, Methods A and B did not use filtration, avoiding WDC loss and resulting in higher WDC quantities. Method A extracted 0.21–0.36% WDC (fresh weight basis), which was higher than Method C but lower than Method B. The lower quantity for Method A compared to Method B was likely due to the application of a centrifugation step after sedimentation, which further separated < 1000 nm colloids from larger particles. Method B, which relied solely on sedimentation for separation, achieved the highest WDC quantity (0.64–1.96% fresh weight basis), possibly because of the incomplete separation of colloidal particles, resulting in the inclusion of larger particles in the extract. This lack of an initial separation step in Method B may have contributed to the higher WDC yield, albeit with reduced selectiveness. Statistical analysis revealed that different extraction methods have a significant influence on the quantity of WDC obtained from soil samples. Notably, the choice of dispersant (e.g., water or electrolyte solution) had no observable effect on WDC yields across all three soil samples and within the method applied, suggesting that the extraction method itself played a more critical role in determining the quantity of extracted WDC.

In general, soils often exhibit variable particle densities because they are a complex mixture of organic and mineral components, whereby minerals are generally heavier than organic constituents. It is known that even slight differences in particle densities can influence the effectiveness of subsequent separation steps, specifically sedimentation and centrifugation, as Stokes' law depends on the particle density. In this study, the calculations of settling times for sedimentation and centrifugation steps using Stokes' Law assumed average particle densities and spherical-shaped particles for the separation of the colloidal fraction from the soil matrix. Consequently, these assumptions may have introduced a bias in the cutoff of different colloid sizes and can lead to underestimation or overestimation of the WDC quantity.

Colloidal particle size distribution and quality

Light scattering analysis revealed that the PSD of the extracted WDC for Method C consistently fell below the 1000-nm cutoff across all soil samples, with the average maximum colloidal particle size of 638 nm (Fig. 3c). This limitation possibly explained the lower WDC yields for Method C and indicated a loss of important information regarding the characteristics of soil colloids, such as the potential alteration of the colloidal PSD as smaller particles dominate the extracted fraction. In contrast, Method B, which relied solely on sedimentation to separate WDC, exhibited the highest proportion of particles exceeding the 1000-nm cutoff (4.0–33.0%). These larger particle sizes could be explained by minor losses of larger particles that might otherwise be lost to filtration and/or separated by centrifugation, and by the uncertainty in calculating the time required to separate the particles, which was generally based on mean raw particle densities. Therefore, using this method could result in the inclusion of larger particles in the separated colloidal fraction.

Using dispersants with varying ionic strengths also influenced the PSD of the soil suspension obtained from the organic soil sample. Dispersing in water yielded a narrower PSD, while the electrolyte solution yielded a broader one when using Method B (Fig. 3c). Electrolytes reduce the repulsive forces between particles, thereby encouraging aggregation or flocculation [58, 59]. Additionally, sedimentation as a separation technique results in higher initial particle concentrations, which promotes the aggregation growth [58]. Consistent with this, a higher proportion of particles greater than < 1000 nm was observed when using the electrolyte dispersant than water. Broadening of the colloidal PSD may also be due to the formation of smaller aggregates [60].

Interestingly, an opposite trend was observed with Method A (Fig. 3c). This method combined sedimentation to isolate < 1000-nm particles with a subsequent centrifugation step to further refine the colloidal fraction. During sedimentation using an electrolyte, flocculated particles were more likely to form spontaneously [12, 61]. However, the centrifugation step appears to effectively separate these flocculated particles, resulting in a narrower PSD compared to the results obtained from the water dispersion. For Method A, the proportion of particles slightly exceeding the 1000-nm cutoff (0.5–3.5%) was lower than that of Method B, but still higher than Method C. This balance suggested that Method A provides the best compromise among the three methods, offering improved precision in WDC measurements.

The findings highlight the trade-offs inherent in different WDC extraction methods, particularly between achieving particle size precision and maintaining WDC yields. Method A, with its use of both sedimentation and centrifugation, emerges as a balanced approach because it effectively extracts WDC while minimizing both the loss of colloidal particles and the inclusion of larger particles in the colloidal dispersion.

Influence on the elemental composition of WDC

To evaluate how different extraction methods and types of dispersants used influence the chemical composition of WDC (< 450 nm), the main elemental components of colloids (C_{org}, Si, Al, and Fe content) were analyzed from the WDC extracted from three soil samples. A high colloidal C_{org} was observed in the organic soil, while lower concentrations were found in soils from organo-mineral and mineral soils (Fig. 4a). The choice of the extraction method significantly influenced the colloidal C_{org} content of the organic soil but did not significantly influence that of the organo-mineral and mineral soil samples. However, the type of dispersant used had a notable impact on C_{org} concentration of the extracted WDC across all three soil samples (Table S6). These observations suggest that ionic strength plays a critical role in mobilizing colloidal organic matter. Dispersing in electrolyte solution was found to potentially increase the concentration of colloidal C_{org}. An increase in ionic strength can inhibit colloid release due to the suppression of the double layer, which leads to a decrease in the repulsive negative surface charge. Cation bridging can also play a role in colloid aggregation, wherein cations form a bridge

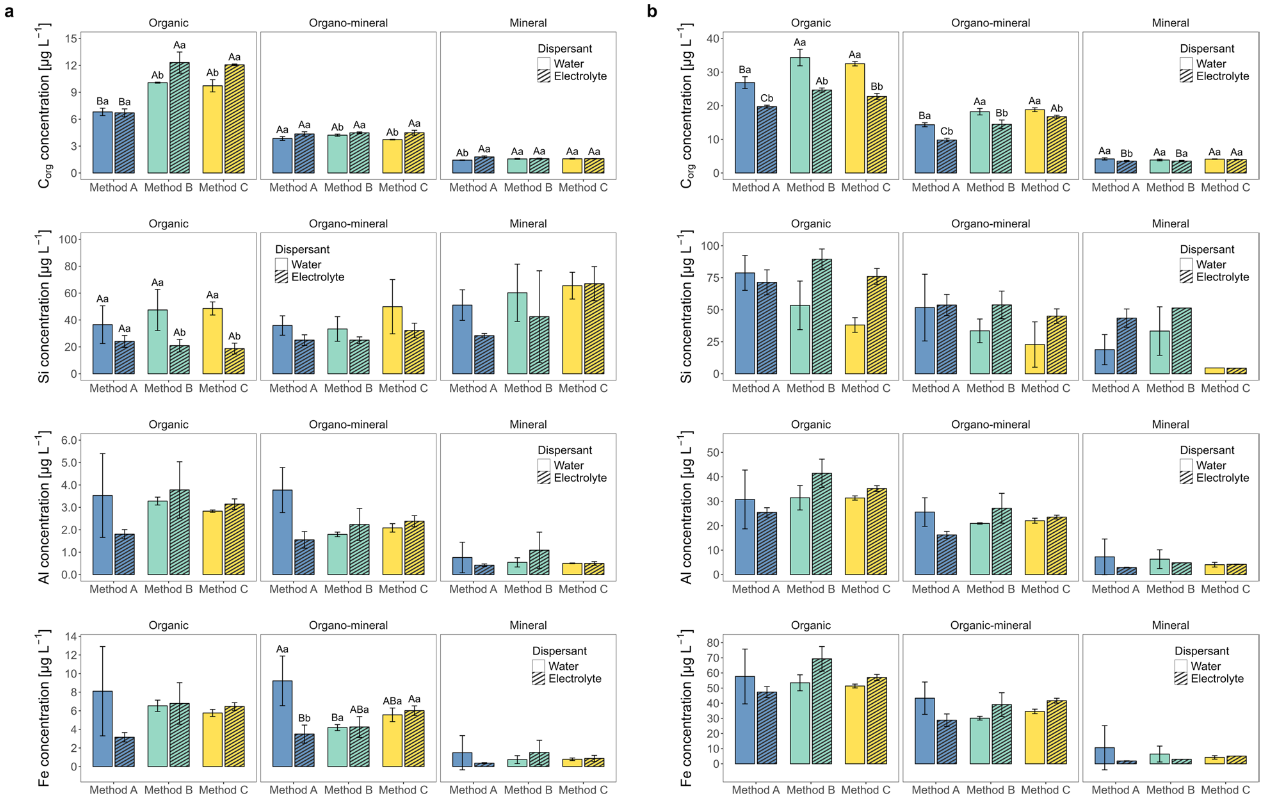

Fig. 4 **a** Colloidal (1 kDa—450 nm) and **b** dissolved (< 1 kDa) concentration of the main elemental component of WDC for organic, organo-mineral, and mineral soil. Capital letters indicate comparison of using different methods within the same dispersant; small letters indicate comparison between dispersants within the same method; different letter designations indicate statistical difference at $p = 0.05$ using a pairwise t-test. The bars without letters indicate no significant effect

between negatively charged particles and organic matter. However, the observed high concentration of colloidal C_{org} may be due to the interparticle steric repulsion and electrostatic stabilization as a consequence of the adsorption of negatively charged organic matter on colloid surfaces [21].

High colloidal Si content was observed in organo-mineral and mineral soils, while lower concentrations were noted in the organic soil. The choice of dispersant also significantly influenced colloidal Si concentration, particularly in the organic soil, whereby dispersing in water resulted in higher colloidal Si concentrations. Si is a key element in phyllosilicate clay minerals, which are dominant inorganic colloids in most soils and carry net negative charges [62]. By preserving these negative charges, dispersants with low ionic strength, i.e., water, can promote colloidal stability and mobility [21, 63], which explains the higher colloidal Si content. The use of an electrolyte dispersant, however, had the opposite effect, as electrolytes promote ion exchange, where counterions replace the acidic hydrogen of silanol groups (≡Si–OH) on the silica surface. This ion exchange reduces the hydration of the silica surface, decreasing colloidal Si stability [64]. The electrolyte dispersant used in this study consisted of both divalent (Ca^{2+}, Mg^{2+}) and monovalent cations (K^+). These cations, especially divalent cations, can compress the electrical double layer and enhance electrostatic shielding [65, 66]. The resulting reduction in electrostatic repulsion promotes the aggregation and sedimentation of soil particles [67, 68], leading to lower apparent colloidal Si concentration in suspension. These findings emphasize the importance of solution chemistry in colloidal Si mobilization, especially in soils with significant organic matter content.

The observed interaction effect of extraction methods and the type of dispersants on colloidal Al and Fe suggests that the effectiveness of the extraction method to extract colloidal Al and Fe may be influenced by ionic strength, and the influence of ionic strength may also be affected by the choice of extraction method (Table S6). In addition, this interaction effect was soil-group dependent and was specifically evident in the organo-mineral soil sample and not in the organic and mineral soils. This likely reflects the stronger role of Fe– and Al–organic matter associations in organo-mineral soils, where mineral phases and soil organic matter interact closely to influence colloid mobilization [69]. Beyond methodological effects, it should be considered that Fe and Al form strong complexes with soil organic matter. These associations act as cementing agents that stabilize organic matter within aggregates and contribute to long-term C sequestration [44]. Such interactions can also influence the composition and mobility of WDC, since Fe– and Al–soil organic matter complexes modify particle surface charge and aggregation behavior. Using Method A, which involves a sedimentation followed by a centrifugation step, resulted in higher colloidal Fe using water in comparison to electrolyte solution (Fig. 4a). Increased ionic strength leads to partial neutralization of repulsive charges and a decrease in activity of Fe species [21, 70], resulting in the coagulation of Fe-bearing colloids. Aggregated colloidal particles are less likely to be effectively separated by separation steps, leading to lower concentrations of colloidal Fe extracted. The significant interaction effect of extraction method and choice of dispersant suggests careful consideration of both method choice and ionic strength in investigating colloidal Al and Fe. The results further emphasize the importance of considering the interplay of both factors in determining the elemental composition of WDC.

Influence on the elemental composition of the dissolved fraction

The amount of C_{org} in the dissolved fraction was influenced by both the extraction method and the dispersants, especially in organic and organo-mineral soils (Fig. 4b). This finding indicates that both factors played a role in determining the quantity of soluble organic matter. Notably, using an electrolyte dispersant resulted in a decrease in dissolved C_{org} concentrations compared to using water in both soils. High ionic strength can lead to a reduction in the amount of dissolved C_{org} that remains dissolved [71]. Additionally, the interplay between the extraction method and dispersants significantly influenced the dissolved Si concentration in the organic soil. The dissolved Fe and Al concentrations, particularly in organo-mineral soil, were also markedly affected by specific combinations of extraction methods and dispersants.

Overall, both extraction methods and dispersants could influence the mobilization of colloidal and dissolved elements. However, their effects varied depending on the specific element and the type of soil material analyzed. For instance, using different dispersants was particularly critical for mobilizing colloidal C_{org} and Si, especially in the organic soil sample. These observed trends suggested that a complex interplay between extraction procedures and dispersants influences colloid-facilitated mobility of elements.

It is important to note that < 450-nm extracts were used for the comparison of the extraction methods based on the elemental composition of the extracted WDC. This size limitation may affect the comparability of elemental composition among the three extraction methods, as the proportion of < 450-nm particles differs between them. Nonetheless, this fraction is considered more reactive [72] and used by several colloidal studies to evaluate the colloidal elemental composition [30, 73, 74]. While it is also important to consider the differences in the proportion of the < 450-nm particles in the PSD of the WDC obtained from the three methods, PSD does not necessarily correlate with the elemental distribution, as different elements can be preferentially associated with colloids of different sizes. For example, C_{org} [72, 75], Fe [75, 76], and Al [75] exhibit higher enrichment in fine colloidal or nanoparticulate fractions. Hence, although focusing on the < 450-nm fraction does not fully reflect all size-dependent variations, it provides meaningful insights into the composition of the most environmentally relevant fine colloidal fraction. Including a broader colloidal size range in future studies would allow for improved comparability among extraction methods.

Feasibility evaluation of the extraction methods

The radar plot shown in Fig. 5 highlights the distinct strengths and weaknesses of each WDC extraction method in terms of cost, time, effectiveness, and precision. The score for the time was determined based on the duration of the conducted method, with shorter extraction times having higher scores. The scoring for cost, effectiveness, and precision was calculated using a simple metric to pragmatically compare the extraction methods. For the cost, scoring was carried out based on whether essential instruments or materials for WDC extraction were required by a given method, as the usage of more instruments or materials indicates additional cost. High scores for cost were assigned to the methods requiring less equipment or material. The score for effectiveness was based on whether the method effectively extracted the colloidal fraction, with consideration of whether there are co-extractions of larger particles or loss of colloidal particles during extraction. The effectiveness score was calculated by determining the difference in the proportions of particles < 1000 and > 1000 nm in diameter from the colloidal PSD. The maximum particle under the < 1000-nm fraction was considered in the effectiveness score calculation by multiplying it by the proportion difference to ensure that methods yielding WDC far below the < 1000-nm cutoff do not appear more effective. The precision score was determined based on

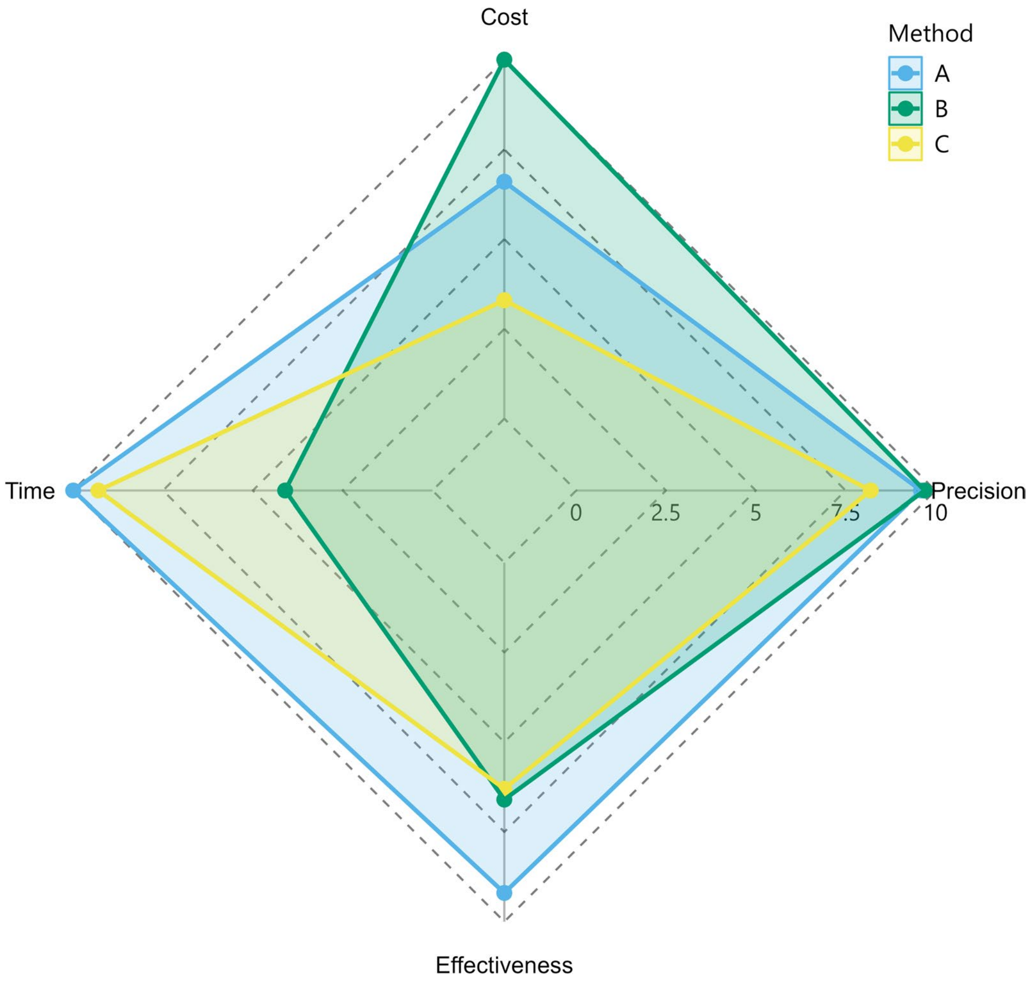

Fig. 5 The feasibility scores (0–10, 10 as the best) of the three selected water-dispersible colloid extraction methods in terms of cost, time, precision, and effectiveness

the CV of the WDC yield obtained for each method, with a lower CV receiving a higher score and indicating better reproducibility. Details on the calculation values are presented in Table S7.

Method A showed the highest scores for time and effectiveness compared to the other methods, and high precision in extracting WDC. However, it scored low in terms of cost, indicating that while it was the most efficient for rapidly extracting < 1000-nm WDC, it could also be a less cost-effective method. The duration of the extraction process was also an important factor, given the intrinsic instability of colloidal suspensions, as coagulation processes and microbial activity could alter the properties of colloidal suspensions, such as PSD, during prolonged processing [50, 55]. Therefore, extraction methods should be as simple and short as possible [10], especially during the separation of WDC dispersion from the bulk suspension.

Method B achieved the highest scores for cost and precision but the lowest for time, emphasizing its suitability for scenarios where cost efficiency and precision take precedence over time constraints. On the other hand, Method C scored well in terms of time but performed poorly in cost and effectiveness compared to the other two methods. Its lower effectiveness makes it less ideal for applications where accurately extracting < 1000-nm colloids is necessary.

Overall, the results indicate that no single method achieved optimal performance across all evaluated criteria. Therefore, the choice of the most suitable extraction method still depends on the specific goals of the research, requiring a balance of cost, time, effectiveness, and precision based on the desired outcomes.

Conclusions and recommendations

The PCA of methodological data from empirical studies showed a diversity of experimental conditions used to extract WDC. Despite the constraints on data availability from some studies, analysis on available data was still useful in exploring existing methodological trends and highlighted the necessity for complete reporting of methodological information for better data reproducibility and comparison across studies.

The experimental comparison of three distinct extraction methods identified through PCA and K-means clustering showed that different extraction methods played a more critical role in gravimetric quantification of WDC in contrast to the choice of dispersant (water or electrolyte solution). With regards to the extraction methods, differences in shaking parameters have less effect on the PSD of suspension taken after shaking of each soil, indicating less influence on the WDC mobilization. The findings suggest that the employed separation techniques for each method influence the effectiveness of extracting the WDC dispersion. Loss of colloidal particles is more prominent when using a filtration step, resulting in lower WDC yield. On the other hand, using sedimentation alone to separate WDC can increase the likelihood of the inclusion of larger particles into the dispersion and may result in the overestimation of WDC yield. The method (Method A) that uses sedimentation followed by a centrifugation step to minimize inclusion of larger particles is therefore recommended to extract WDC from soils.

This study also demonstrated the influence of using different extraction methods and dispersants on the elemental composition of WDC extracted from three different soils. The ionic strength of dispersants played a critical role in mobilizing colloidal C_{org} and Si, particularly in organic and organo-mineral soils. Complex interaction between the choice of dispersant and extraction method influences the colloidal and dissolved Al and Fe concentrations in organo-mineral soils.

From the feasibility comparison, none of the three extraction methods performed the best in all criteria, which are cost, time, precision, and effectiveness. Nevertheless, we recommend Method A, which involves a short shaking time followed by a two-step separation (sedimentation and centrifugation step) in extracting WDC, as it performed well in three out of four criteria. Database analysis also showed that this method had been used in extracting WDC in mineral soils with a wide range of soil properties and from a variety of land uses and climates. Further testing of this extraction method with soils from different land uses and soil types is still needed.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s12302-025-01279-3.

Supplementary material 1.

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Author contributions
J.C.A.C. conceptualized the study, wrote the main manuscript, performed the experiments, analyzed the data, and prepared all the figures. A.D.M and R.C. wrote part of the manuscript. J. K., A.E.B, L.W., and N.S. conceptualized the study and supervised the work. All authors reviewed the manuscript.

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Data availability
No datasets were generated or analysed during the current study.

Declarations

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Not applicable.

Consent for publication
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Competing interests
The authors declare no competing interests.

Author details
[1]Institute of Bio- and Geosciences, Agrosphere (IBG-3), Forschungszentrum Jülich GmbH, Wilhelm-Johnen-Straße, 52425 Jülich, Germany.

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