Study of UV–vis responses of mineral suspensions in water

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Abstract

The aim of this work is to study UV–vis responses of mineral particles and colloids in order to obtain a better understanding of "physical" absorption phenomena. The study of mineral-suspended solids and colloids allows to point out the influence of the size on UV–vis spectra. A typology of UV responses of particles according to their size and a model spectrum of mineral colloids are proposed.

Keywords: Colloids; Particles; UV–vis spectrophotometry; Mineral suspensions

1. Introduction

Suspensions are commonly found in many industrial fields such as pharmaceutical industry, food industry, materials, wastewater and water treatment either in intermediate products form or in effluent form. The characterization of suspensions is necessary in order to optimise and to control processes, separation treatments (filtration, flotation, hydraulic sizing, ...) as well as treatment processes. For example, in wastewater treatment, physical and biological processes are influenced by heterogeneous fractions (settleable, supra-colloidal and colloidal fractions), particularly for dispersion and agglomeration phenomena. Therefore, the understanding of these phenomena, the development of methods allowing the characterization and the quantification of solids in sewage are necessary for the optimisation of treatment processes and for the evaluation of their performance [1,2].

Optical methods are often chosen for the control of water quality because they can be used on line and allow a real time monitoring. They are based on optical phenomena caused by the interaction between light and particles. Among optical methods, turbidimetry based on absorption and scattering properties of suspended solids is often used to estimate total suspended solids (TSS) concentration. But turbidity depends on concentration, size and surface properties of particles [3,4] and the measurement by diffusion considers mainly the colloidal fraction [5] limiting its use to wastewater of constant quality. However, a new method to extract particle concentration and a characteristic particle size from turbidimeter readings has been developed [6]. The method requires a turbidimeter capable of measuring the forward (12°) and sideways (90°) scattered light simultaneously. Tested on the calibration of filter aids, an industrial pigment and yeast, the method is limited to the range of particle concentration (1–200 ppm) and characteristic particle size (1–100 μm) used in the calibration. A second limitation arises from the fact that the other parameters influencing scattering (optical properties, particle shape and porosity, width of the size distribution) are not considered.

Another one is UV spectrophotometry which integrates different combined optical phenomena such as physical absorption (particles' absorption, diffusion, refraction, diffraction) and chemical absorption. That is the reason why optical responses of wastewater are complex and difficult to interpret.

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More precisely, when a particle is illuminated by a beam of light, it sends back light in all directions of the space: it is the diffusion phenomenon whose three components are the refraction, the reflection and the diffraction [7]. In some cases, the light may be absorbed by particles. These interactions between a light beam and particles depend mainly on the ratio between the particle size (diameter d) and the wavelength (λ) of the beam of light. In order to clarify the domain of each phenomenon, a size parameter α is generally used [7,8]:

$$\alpha = \frac{\pi d}{\lambda}$$

According to the value of the parameter α, and with light sources usually used (UV, visible, near IR), three domains of diffusion are considered:

- For $\alpha < 0.3$, i.e. for particles smaller than the wavelength, the optical model of the Rayleigh diffusion is used. Particles diffuse light as much forward as backward.
- For $\alpha > 30$ which corresponds to particles whose size is greater than the wavelength, laws of geometric optics and diffraction are used. In this case, light is diffracted, meaning that the diffusion is mainly concentrated in front of the particles.
- For $0.3 < \alpha < 30$, i.e. for particles whose size is close to the wavelength, this intermediate domain corresponds to the validity limit of the last models (diffusion and diffraction), because diffusion is also influenced by reflection, refraction and absorption phenomena. These can be taken into account by the complex theory of Lorenz–Mie. Optical properties of particles are necessary and optical model with real and imaginary parts of refractive index must be used.

These domains of diffusion are valid for spherical particles and single diffusion. About the shape of the particles (spherical in Mie model), it is well known that it has a strong influence on optical properties [9] and other approaches have been proposed for non-spherical particles [10,11]. The shape but also the orientation of the particle is important. Thus, a study [12] has shown a strong effect of particle shape on the particle size distribution, measured by laser diffraction and scattering and by photosedimendation, because of particles' orientation. In the case of aggregate, the relation between monomer size and its number in the aggregate determines the optical properties of the particles. Extinction observations are used for estimating the size of scattering particles. However, if the particle size is comparable to the wavelength of light, the extinction will depend on particle shape. For aggregate particles with a size comparable to the wavelength, the spectral dependence of extinction efficiency is less steep than that for equivalent spheres, and its maximum is shifted to larger size parameters, i.e. smaller wavelengths [13].

Light–particle interactions have been intensively studied in relation to the development of granulometric methods (laser granulometry, for example) [9]; however, little is known about the UV–vis response of particles. UV–vis spectrophotometry so far focussed mainly on characterization and analytical determination of organic compounds [14,15]. We can note that Fu et al. [16] have studied the optical response (in UV–vis domain) spectra of organic pigments in aqueous dispersions. They have clearly shown that the optical response is the result of absorption and scattering of light by the pigment particles. The intensity of the two phenomena (absorption and scattering) is used to determine the dispersion degree of organic pigments. Alupoaei et al. [17] have proposed a model for the quantitative interpretation of UV–vis spectra of prokaryotic microorganism suspensions (that present some specific absorption bands). Their model is based on light scattering theory, spectral deconvolution techniques, and on the approximation of the frequency-dependent optical properties of the basic constituents of living organisms. Thomas et al. [14,15] have proposed reference spectra of suspended solids and colloids to model the interferences of UV spectra of wastewater and natural waters.

On the other hand, Azéma et al. [18] have shown the influence of granulometric size distribution on the UV responses of organic solids but the physical response representing no chemical absorbance has not been studied.

The aim of this work is to study UV–vis responses of model particles in order to obtain a better understanding of "physical" absorption phenomena. Model particles chosen are of mineral nature in order to avoid chemical absorption. The influence of the size of particles is particularly studied in order to propose a typology of UV–vis response of particles.

2. Experimental procedure

2.1. Samples

The particles studied are mineral suspensions in slurry or in powder form. Table 1 gives the origin of the product and the initial concentration of samples for UV spectra acquisition. The slurry formulation may contain additives differing in chemical nature and properties. Table 2 gives some properties of studied minerals (shape, shape factor and density). Main granulometric characteristics of studied suspensions are summarized in Table 3.

Two types of particles are particularly studied by UV–vis spectrophotometry, particles whose size is larger than 1.2 μm approximately, usually called total suspended solids in water and wastewater, and colloids whose size ranges from 0.1 to 1.2 μm. The calculation of the size parameter for UV–vis wavelengths (from 200 to 800 nm) allows determining the diffusion domain of TSS (>1.2 μm), "larger" colloids (0.1–1.2 μm) and "soluble matter" (<0.1 μm) approximately (Fig. 1):

- for colloids, the light diffusion corresponds to the Mie domain;

Table 1
Origin and concentrations of studied samples

Mineral suspensions	Origin	Initial concentrations (for UV spectra acquisition)
Carbonate (slurry)	Les papeteries Lancey (France)	250 mg L^{-1}
Kaolin (slurry)	Les papeteries Lancey (France)	250 mg L^{-1}
Talc (slurry)	Les papeteries Lancey (France)	10 g L^{-1}
"Amazone 88" kaolin (powder)	Westerlund Corporation	207 mg L^{-1}
"Ultracarb" Carbonate (powder with stearate coating)	Microfine Minerals Ltd.	206 mg L^{-1}

Table 2
Properties of studied minerals (adapted from [20,21])

Mineral suspensions	Shape	Shape factor[a]	Density
Carbonate (slurry and powder)	Rhombus	1–7	2.7
Kaolin (slurry and powder)	Hexagonal plaques	1–2	2.6
Talc (slurry)	Flakes	1–2	2.6–2.8

[a] Ratio between length and central width (determined from microscopic appearance).

Table 3
Main granulometric characteristics of studied suspensions (from [21,22])

Suspension	Main modes (μm)
Talc	10
Kaolin (slurry)	0.6 and 2
Carbonate (slurry)	0.5 and 2
Kaolin (powder)	5 and 50
Carbonate (powder)	10 (broad granulometric spectrum)

- for TSS, the light diffusion can correspond to the Mie domain or the diffraction domain in accordance with the size.

2.2. Fractionation technique

The experimental procedure used to separate particles in suspensions is described in Fig. 2. In this study, the particles are separated by filtering with glass fiber filters (approximately 1.2 μm). Colloids are separated from "soluble" matter by crossflow-microfiltration (0.1 μm) on acetate membrane. A filtration (on nylon filter) at 10 μm is used to isolate the larger particles (coarse) in the studied suspensions.
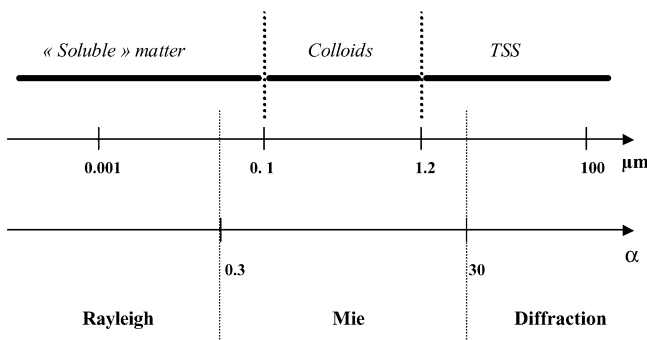

Fig. 1. Diffusion domain according to the size of particles.

2.3. UV–vis spectrophotometry

2.3.1. Spectrophotometer

The UV spectra study concerns the acquisition of absorption spectra, i.e. the variation of absorbance value with wavelength. The spectrophotometer used is an Anthelie Light (Secomam) of 2 nm bandwidth, with a quartz cell of 10 mm pathlength. A speed of 1800 nm/min is chosen with respect to the presence of suspended solids in samples. Dathelie PC software (Secomam) is used for data processing. The influence of settling on the acquisition of data was studied for suspensions prepared from powder. The measured errors on the absorbance values are the following:

- 1.2% for kaolin suspension;
- 0.75% for carbonate suspension;
- 6.0% for talc suspension.

2.3.2. Exploitation methods of UV–vis spectra

- The first method of exploitation of UV–vis spectra is the difference of spectra, corresponding to the difference between the absorbance values for each wavelength [19]. It is thus possible to visualise the spectra of different fractions of one sample. For instance, the spectrum of suspended solids corresponds to the spectrum of the raw sample minus the one of the sample filtered at 1.2 μm, with the following relation for each wavelength:

$$\text{ABS}_{\text{TSS}}(\lambda) = \text{ABS}_{\text{Raw}}(\lambda) - \text{ABS}_{\text{Filtered}}(\lambda),$$

$$\text{with ABS}_{\text{Raw}}(\lambda) > \text{ABS}_{\text{Filtered}}(\lambda)$$

The difference between the spectrum of the 1.2 μm filtered suspension at 1.2 μm and the filtered suspension at 0.1 μm allows one to obtain the spectrum of colloids.

- The second one is the normalisation of spectra in order to compare the spectra shape from a qualitative point of view. Normalised spectra have the same area, i.e. the same total sum of absorbance values inside the wavelength window [19].

For a given spectrum and a small acquisition step (e.g. 1 nm), the area is:

$$\text{AREA} = \sum_{\lambda=200}^{350} A(\lambda) = C\ell \sum_{\lambda=200}^{350} \varepsilon(\lambda)$$
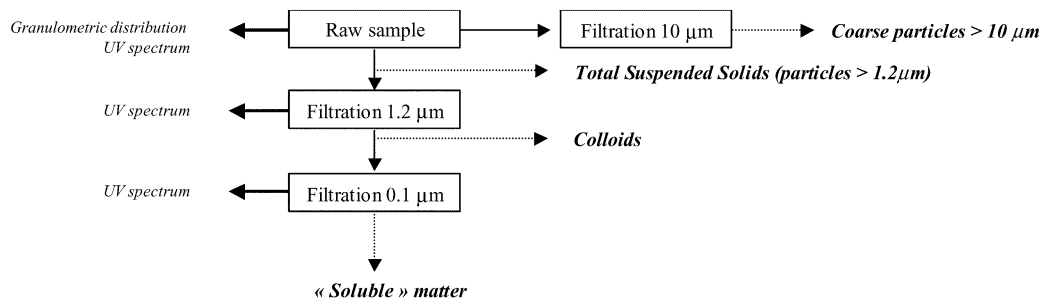
Fig. 2. Fractionation technique.

Table 4
Optical models used by laser granulometry

Suspension	Refractive index real part	Refractive index imaginary part	Model
Talc	1.65	0.1	Mie
Kaolin (powder and slurry)	1.65	0.1	Mie
Carbonate (powder and slurry)	1.48	0.1	Mie

where C is the sample concentration, ℓ is the optical path-length, and ε is the absorptivity at the wavelength λ.

Normalisation of the spectrum, according to a given value of the norm A_N, changes the absorbance value at the wavelength λ, $A_i(\lambda_i)$, into a normalised absorbance, $A_{iN}(\lambda_j)$:

$$A_{iN}(\lambda_j) = \frac{A_i(\lambda_j)A_N}{\sum_{\lambda_j} A_i(\lambda_j)h}$$

with $A_{iN}(\lambda_j)$: value of the absorbance at the wavelength λ_j after normalisation (a.u.); $A_i(\lambda_j)$: value of the absorbance at the at the wavelength λ_j before normalisation (a.u.); A_N: relative area of normalisation (fixed value of, for example, 100 (a.u.) nm; h: step of 1 nm.

2.4. Laser granulometry

Measurements of particle size distribution are performed with a LS 230 Beckman-Coulter laser granulometer. The volume of the analysis cell is 125 ml. The incident light is transmitted through the suspension that circulates. The Fraunhofer model considers the particles as spherical and opaque, and allows calculating granulometric distributions of suspensions in terms of volume percentage. Nevertheless, the refractive index of particles composed of a real part and an imaginary one, which corresponds to absorption, has to be taken into ac-

count in the Fraunhofer model, as small particles involve diffusion and transparent particles involve refraction phenomena. Optical models are presented in Table 4.

3. Results and discussion

Several parameters such as diffusion domain (Rayleigh, Mie or diffraction), the type of diffusion (single or multiple) and the influence of the size of particles (colloids or TSS) are studied.

3.1. Determination of the diffusion domain of mineral suspensions

The granulometric characteristics of mineral suspensions studied are estimated by laser granulometry. The main granulometric modes are presented in Table 3. The talc suspension is composed of TSS predominantly whereas carbonate and kaolin suspensions (prepared from slurry) contain colloids and TSS.

The knowledge of main granulometric modes of suspension allows to calculate the size parameter for 200, 400 and 800 nm and to deduce the main domain of light diffusion in the UV–vis region (Table 5):

- concerning TSS of talc, the diffusion domain is the diffraction one;
- concerning TSS and colloids of carbonate and kaolin suspensions, the diffusion domain is the Mie domain.

First of all, TSS of talc whose size is greater than the wavelength (diffraction domain) are studied. Then, results related to colloids and TSS of kaolin and carbonate whose size is close to the wavelengths (Mie domain) are presented.

Table 5
Size parameter for studied suspensions

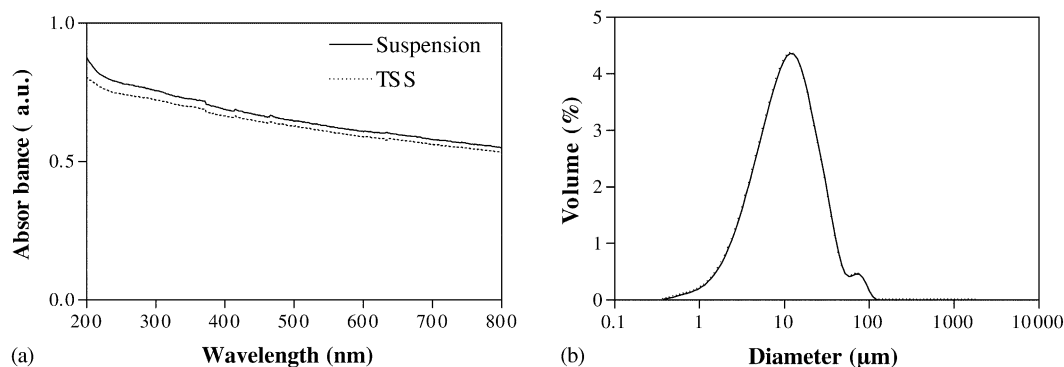|	Diameter; nature of the particles			
	0.6 μm; kaolin (slurry)	0.5 μm; carbonate (slurry)	2 μm; kaolin/carbonate (slurry)	10 μm; talc, kaolin/carbonate (powder)
α for $\lambda = 200$ nm	9.4	7.8	31.4	157.1
α for $\lambda = 400$ nm	4.71	3.9	15.7	78.5
α for $\lambda = 800$ nm	2.35	3.0	7.9	39.3
Diffusion domain	Mie, $0.3 < \alpha < 30$	Mie, $0.3 < \alpha < 30$	Mie, $0.3 < \alpha < 30$	Diffraction, $\alpha > 30$

Fig. 3. UV–vis spectra of a talc suspension ($10\,\mathrm{g\,L^{-1}}$) (a) and granulometric distribution (b).
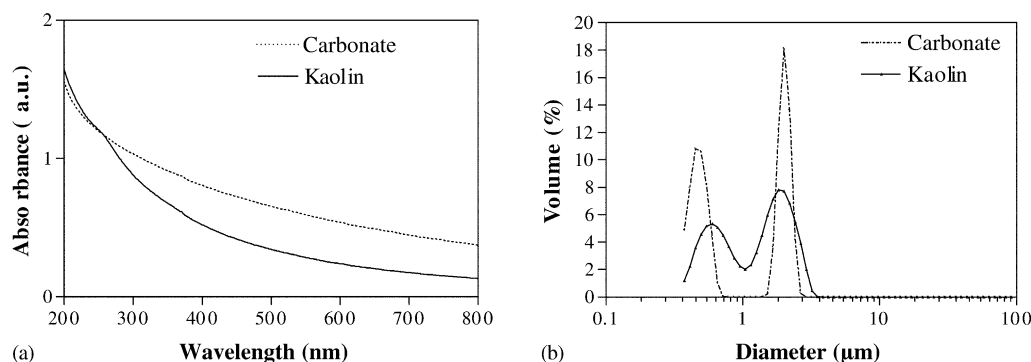
Fig. 4. UV–vis spectra of carbonate ($206.3\,\mathrm{mg\,L^{-1}}$) and kaolin suspensions ($207\,\mathrm{mg\,L^{-1}}$) (a) and granulometric distribution (b) prepared from slurry.

3.2. Optical response of talc suspension

Fig. 3 presents the UV–vis response and the granulometric size distribution of a suspension of talc. The suspension is mainly composed of TSS and represents a main granulometric mode at $10\,\mu\mathrm{m}$ and a broad distribution at about $100\,\mu\mathrm{m}$. The UV–vis response due to diffraction is only slightly dependent on wavelength.

3.3. Optical responses of kaolin and carbonate suspensions

3.3.1. UV–vis responses of suspensions

Fig. 4 presents the UV–vis responses and the granulometric size distributions of kaolin and carbonate suspensions (prepared from slurry). We can compare these suspensions as they are characterized by similar refractive index and granulometric distribution (Tables 2 and 4). The absorbance values decrease according the wavelength. After 250 nm, although the concentrations are similar, the absorbance values of the carbonate spectrum are higher than the kaolin spectrum ones. This result can be due either to the presence of finest colloids ($<0.45\,\mu\mathrm{m}$) in the carbonate suspension (partly detected by laser granulometry), which diffuse more light, or to a different shape of particles which influence the diffusion of light. The first hypothesis has been validated by a fractionation at $0.45\,\mu\mathrm{m}$ of these suspensions, showing that the carbonate suspension contains more colloids (whose size is lower than $0.45\,\mu\mathrm{m}$) than the kaolin one.

A shoulder at 260 nm can also be shown on the kaolin suspension spectrum. This chemical response is probably due to the presence of an organic compound used for the formulation of kaolin (coating, dispersant or surfactant).

Fig. 5 presents the absorbance values at 400 and 800 nm of UV–vis spectra of suspensions versus concentration. Although the suspensions contain both colloids and TSS, the absorbance is proportional to the concentration, meaning that the diffusion of light is single for the tested concentrations. At 40 nm, the saturation is reached for a carbonate suspension of $500\,\mathrm{mg\,L^{-1}}$ whereas the absorbance value is below 2.0 a.u. for the same kaolin concentration. It confirms the presence of finest colloids in the carbonate suspension. The differences observed between the two suspensions can also be due to the particles' shape, aggregation or refractive index (see Tables 2 and 4).

3.3.2. UV–vis responses of colloids and TSS

Fig. 6 compares normalised UV–vis responses of mineral TSS and colloids (prepared from slurry). Except for the spectrum of carbonate TSS, exhibiting a very broad slight maximum leading to a quasi-independence of absorbance value with wavelength, the other spectra show absorbance decrease from UV to visible region. However, this observation should be confirmed for kaolin TSS, considering the presence of
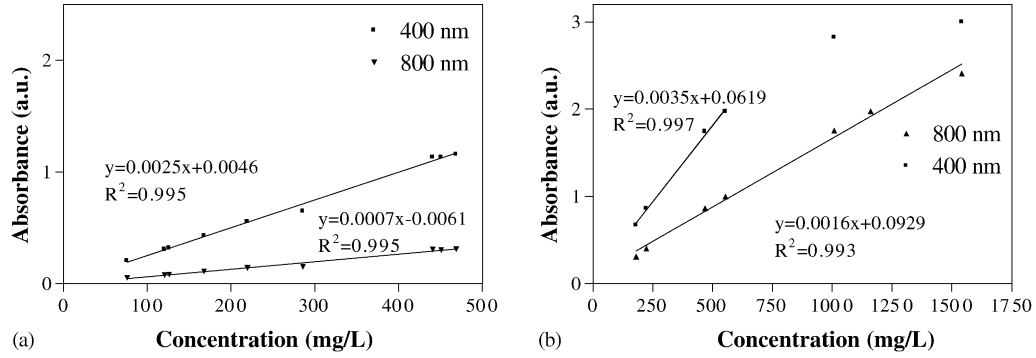
Fig. 5. Absorbance values vs. concentration for (a) kaolin suspension and (b) carbonate suspension prepared from slurry.
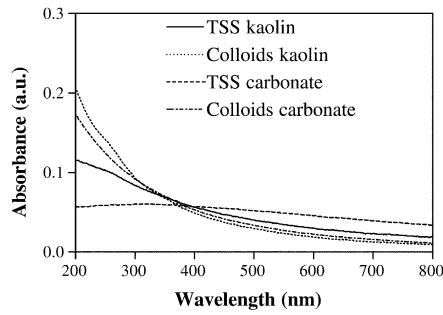

Fig. 6. Normalised UV–vis spectra of TSS and colloids between 200 and 800 nm.

organic contaminant (previously noted) and its possible impact on the spectrum shape.

With respect to spectra shapes, three wavelength domains can be proposed: 200–300, 300–450 and 450–800 nm.

The contribution of absorbances to the total area of the spectrum in these domains is calculated as follows for TSS and colloid spectra:

$$\text{Contribution}_{200-300\,\text{nm}}(\%) = \frac{\sum_{\lambda=200}^{\lambda=300} A(\lambda)}{\sum_{\lambda=200}^{\lambda=800} A(\lambda)} \times 100$$

$$\text{Contribution}_{300-450\,\text{nm}}(\%) = \frac{\sum_{\lambda=300}^{\lambda=450} A(\lambda)}{\sum_{\lambda=200}^{\lambda=800} A(\lambda)} \times 100$$

$$\text{Contribution}_{450-800\,\text{nm}}(\%) = \frac{\sum_{\lambda=450}^{\lambda=800} A(\lambda)}{\sum_{\lambda=200}^{\lambda=800} A(\lambda)} \times 100$$

Fig. 7 shows that:

- The contribution of the range 200–300 nm is more important for the colloid spectrum (40–50% of the total area) than for TSS (20–30% of the total area).
- Contributions of the range 300–450 nm are similar (30% of the total area) for colloids and TSS.
- The contribution of the range 450–800 nm is more important for TSS spectrum (35–50% of the total area) than for colloids (20–25% of the total area).

For a mineral suspension composed of TSS and colloids in the same concentration, the UV response from 200 to 300 nm seems to mainly be due to the colloid one whereas the visible response from 450 to 800 nm could correspond to the TSS one. However, the presence of a shoulder at 260 nm, as noted previously, shows that a validation with kaolin suspensions free of organics is necessary.
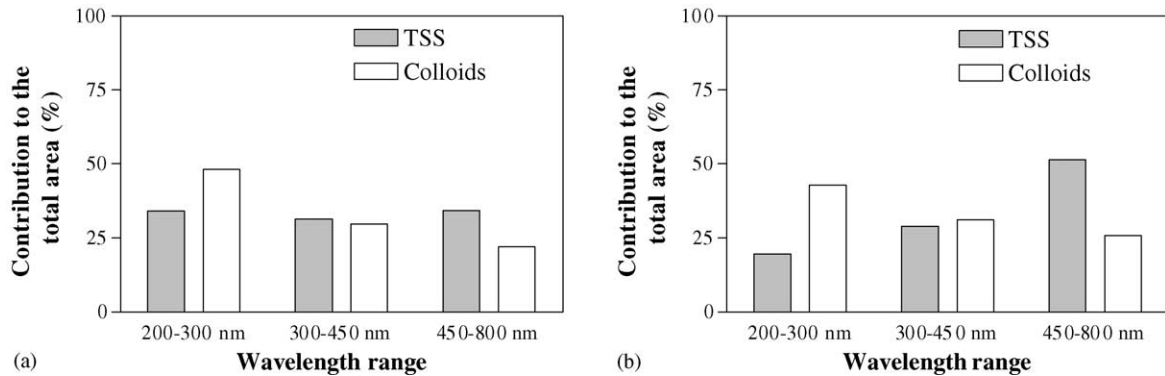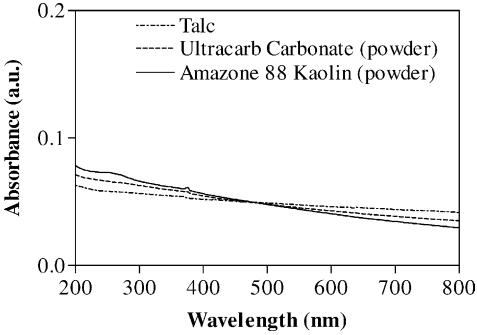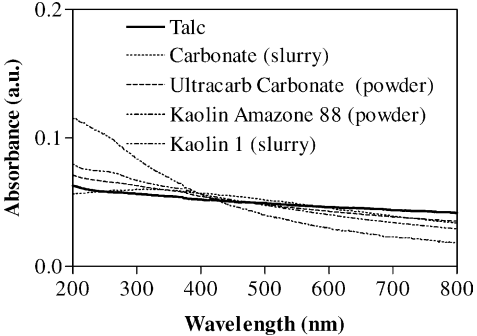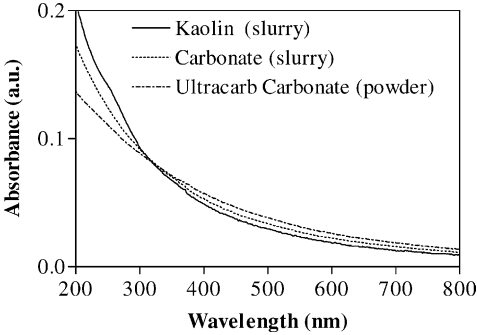

Fig. 7. Contribution of 200–300, 300–450 and 450–800 nm ranges to the total area of TSS and colloid spectra of (a) kaolin and (b) carbonate suspensions.

Table 6
UV–vis responses (normalised spectra) of particles according to their size

Particles	UV–vis spectra	Comments
Coarse particles (>10 μm)		Diffraction domain ($\alpha_{200\,nm}$ = 157 to $\alpha_{800\,nm}$ = 39); absorbance depends very slightly on the wavelength ($A_{200\,nm}/A_{800\,nm}$ = 1–3)
TSS including coarse particles (>1.2 μm)		Mie and diffraction domains ($\alpha_{200\,nm}$ > 19 to $\alpha_{800\,nm}$ > 5); absorbance depends slightly on the wavelength except for Kaolin (slurry) ($A_{200\,nm}/A_{800\,nm}$ = 1.5–6)
Colloids (0.1–1.2 μm)		Mie domain ($1.57 < \alpha_{200\,nm} < 19$ to $0.4 < \alpha_{800\,nm} < 5$); absorbance depends strongly on the wavelength ($A_{200\,nm}/A_{800\,nm}$ = 10–24); $A = a(1/\lambda) + b$; $a \sim 0.03$–0.05; $b \sim 0.03$–0.06

These observations should be confirmed by a comparison of optical responses based on the use of extinction coefficient rather than absorbance value.

3.4. Typology of UV–vis responses of particles according to their size

Table 6 gathers all UV–vis responses of particles according to their sizes:

- For particles whose diameter is larger than 10 μm, the diffusion domain is the diffraction. This phenomenon is characterized by UV–vis spectra with absorbance values slightly dependent on wavelength (diffraction and Mie/diffraction domain) which are almost independent of the wavelength. The ratio between absorbance values at 200 and 800 nm is about 2.

- For TSS (>1.2 μm), the diffusion domains are both Mie and diffraction ones. TSS present the same optical response as particles whose size is greater than 10 μm even if a marked slope is noticed for kaolin. The ratio between absorbance values at 200 and 800 nm is about 2 except for kaolin, because of organic contaminant.

- For colloids, the diffusion domain is the Mie one. UV–vis responses depend strongly on wavelength. The ratio between absorbance values at 200 and 800 nm ranges from 10 to 24.

Moreover, a linear relation between normalised absorbance values and the reciprocal wavelength (Fig. 8) can be proposed to model the UV response of colloids:

- $A = 35.3(1/\lambda) - 0.032$ $R^2 = 0.9976$ (carbonate Ultracarb, powder);
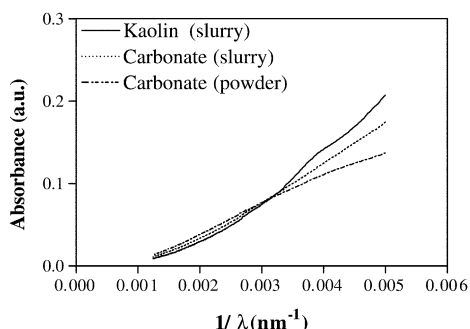
Fig. 8. Normalised absorbance values of colloid spectra vs. $1/\lambda$.

- $A = 42.6(1/\lambda) - 0.048 \qquad R^2 = 0.9916$ (carbonate, slurry);
- $A = 49.4(1/\lambda) - 0.064 \qquad R^2 = 0.973$ (kaolin, slurry).

The difference observed between UV spectra of particles in slurry form and in powder form can mainly be explained by the presence of organic compound(s) used in the formulation which may absorb in the UV region (particularly for kaolin). For range of 200–300 nm ($1/\lambda$ from 0.003 to 0.005), carbonate powder spectrum deviates negatively, and kaolin positively from linearity. Further validation should be made to check the linearity observed.

4. Conclusion

In conclusion, the UV–vis responses of non-absorbing particles can be modelled even if the physical phenomena are complex and the refraction index and the shape of particles have not been taken into account. The study of talc, kaolin and carbonate particles confirms the influence of the size of particles on the optical responses. The physical absorption of suspended solids with size greater than 1 μm is characterized by absorbance slightly dependent on the wavelength. For colloids, a relation between absorbance values and $1/\lambda$ is proposed. The results also show that it is possible to discriminate predominant domains of diffusion by TSS and by colloids, useful for the improvement of the quantification of solids in water and wastewater. However, the method is restricted to samples practically free of UV-absorbing contaminants or at least to samples where the influence of these contaminants can be quantified. Otherwise, the content of colloidal matter will be strongly overestimated.

Acknowledgments

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