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Experiments on depolarized optical scattering to sense in situ the onset of early agglomeration between nano-size soot particles

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Abstract

Soot aerosol particles generated in hydrocarbon flames are investigated in this paper by laser light scattering techniques. The contribution of co- and cross-polarized scattered light, each for vertical and horizontal linear polarization states of the incident laser light at 514.5 nm are measured at variable polar angles θ. Main results here reported are the following: (1) the vertical depolarization ratio ρ_v measured against the scattering angle is very low and flat in the case of smaller chain-like (aggregate fractal dimension $D_f \approx 1.3$), whereas, in the case of larger branched-chain aggregates ($D_f \approx 1.7$), ρ_v exhibits a maximum at about 90° (2) in contrast to the reciprocity theorem, the measured ratio I_{HV}/I_{VH} between the two depolarized contributes for each of the two possible linear (vertical and horizontal) polarization states of the incident light, is found to be different than unity when first sticking of the primary particles occurs. Therefore, the measurement of the reciprocity ratio I_{HV}/I_{VH} seems a powerful and reliable tool to establish the onset of the early aggregation mechanism between nano-size soot particles formed in a flame. © 2002 Elsevier Science Ltd. All rights reserved.

1. Introduction

The in situ investigation about the properties of soot particulate formed in combustion process represents a classic inverse problem for radiative heat transfer in a inhomogeneous medium at high temperature. Thus, it is crucial when experiments are made by optical techniques, to recognize from the measured scattering and absorption characteristics the size, morphology and nature of the agglomerates of fine particles. The phase function and single scattering albedo, which are usually

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measured in the experiments, enter directly in the radiative transfer equation, and, therefore, must to be known to solve the direct problem of radiative transfer in a participating medium [1]. The mechanism of multiple scattering, absorption and emission are not yet well known when ensemble of nano-size soot particles undergo coagulation or sintering in flame environments. Consequently, predictive tools are still far from representing the complexity of physical phenomena. In the field of the electromagnetic integral formulation of scattering theory for aggregates or irregularly shaped particles, some numerical approaches (DDA, IEFS, CED–CEMD) have been extensively applied in the past. Interesting review of such methods are available, see for instance in [2–4], and the references therein cited. Limitation of such models are, beyond the computational effort, the facts that many particles, and not a single aggregate, are generally involved in a radiative transfer process and the morphology of such structures is not a priori known.

The optics of fractal aggregates has been also investigated by the so-called Rayleigh–Debye–Gans (RDG) scattering theory, which was first proposed in the modern literature by Dobbins and Megaridis [5] and, thereafter, by several other authors [6–9]. The applicability of such an approximate approach to coupled scattering/extinction experiments is hindered for weakly absorbing materials. Moreover, RDG formulation still remains disputable, owing to the fact that even for particles with the same nature such as soot, it can be observed enormous variability of scattering and absorption properties during the formation history, the so-called "carbonization" process [10], However, it holds true that RDG approach allows for easy reduction of the angular pattern measurements of vertical polarized scattered light intensity for randomly oriented fractal-like aggregates (FA). In particular, the average form factor of vertical (scattered)–vertical (incident) differential scattering I_{VV} is found to admit two approximate functional forms (Guiner regime and power-law regime), which can be directly linked to the two observable quantities, namely, the radius of gyration and the fractal-like dimension. The limitations of such an approach with respect to the more exact theoretical formulation IEFS [11] is represented by the fact that both the multiple scattering and the self-interaction contributions are considered negligible, that reflects into the fact that RDG-FA formulation does not satisfy the optical theorem. The analysis of the range of validity of RDG-FA with respect to the IEFS formulation has been discussed in the literature [12].

As far as the flame generated agglomerate are concerned, it is well known that soot clusters have typical appearance of branched assemblies of single sub-units, known as primary particles or *spherules*. They are usually characterized in terms of *fractal-like* objects [5,13,14], i.e., obeying to a relation of form $M \propto (2R_g/a_P)^{D_f}$ between aggregate mass M, gyration radius R_g and fractal dimension D_f and spherule size a_P. When the genesis of primary particles is investigated, then the condensable molecules are considered, which very rapidly (with time constant in the order of 1 ms) transform from the gas to the particle phase and the primary particles are sometimes called *monomers*. On the other hand, each single soot spherule that is sized typically in the range 10–40 nm, is usually retained to behavior as a Rayleigh's scatterer at visible wavelengths (see for instance [8] and references therein cited). As a rule of thumb, an upper limit of 10% error is considered with respect to the exact Mie theory when the size parameter of primary particles is smaller than 0.3 [12]. Thus, the angular pattern of I_{VV}, namely of intensity of light scattered with the same vertical polarization with respect to the incident light, in the case of non-agglomerated primary particles is uniform with respect to the scattering (polar) angle θ [15], whereas an enhancement of I_{VV} is expected at near zero scattering angles for a factor that is the squared number of primary particles per cluster [16].

Angular patterns of I_{VV} co-polarized light allows for the direct evaluation of fractal dimension D_f and gyration radius R_g of fractal aggregates by standard procedures reported in many papers [5,17,18]. Fractal dimension gives information about the intra-cluster density [19] by means of the measured pair-correlation function $g(\vec{r})$ ($\vec{r}$ is the position vector between two sites in the same aggregate). From an analytical point of view, $g(\vec{r})$ is the Fourier transform of the I_{VV} angular patterns measured in the experiments [20]. Pair correlation function must contain as factor a *cut-off function* $h(\vec{r}/\xi)$, owing to the finite size of the cluster [19,21], where ξ is known as the *correlation length* of the fractal aggregate [21]. Mass fractal power correlation holds true only in a limited range r of inspection, namely for q between ξ^{-1} and a_P^{-1} [22] where $q \equiv (4\pi/\lambda)\sin(\theta/2)$ is the magnitude of the scattering wave vector. Within this range, the power law for co-polarized scattered contribution $I_{VV}(q) \propto q^{-D_F}$ applies. Nevertheless, fractal analysis is considered sufficiently reliable only for relatively large aggregates, for instance when the ratio between the radius of gyration and the size of single primary particle is at least of order 10 [19]. Thus, one of the most intriguing questions that comes out regarding this approach is the following: how can scattered light to be correlated to the structural features of smaller aggregates composed of some tens of nano-size soot particles? Contributes to this issue has been previously reported by some authors [23] which assume some canonical aggregate shapes and suggest a computationally-based methodology to identify the agglomeration process from multi-wavelength measurements of the scattered light at a few angles. Lack of simple experimental techniques still exists to distinguish the cases of aggregated against not aggregated particles.

The aim of this paper is to report some experimental findings that give evidence about the fact that the depolarized scattered light is effectively a very sensitive tool to investigate such smaller aggregates. In particular, depolarized scattering can be crucial to discriminate the early onset of the aggregation mechanism between isolated nanoparticles. This is not a trivial issue to be addressed, especially in the case of very small clusters (e.g., dimers, trimers, etc.), for which the overall dimension of aggregates still lies in the Rayleigh regime.

2. Experiments

The experimental set-up is sketched in Fig. 1 and described in detail in previous work [24] that was dedicated to demonstrate the feasibility of an experimental method to obtain without any sampling the size of soot primary particles in flame aggregates by a FFT inversion technique applied to the measurements of angular scattering patterns.

An Ar-Ion laser beam at 514.5 nm is mechanically modulated at 931 Hz, and is used to probe soot particles generated within different flames obtained by a Bunsen burner (1 cm inner diameter, air-inlet blocked) fuelled with different hydrocarbon gases (propane and ethylene, purity better than 99.95%). The scattered light is collected at angles θ between 10° and 160° by a rotating optical system composed by an interference filter (half-width spectral response 1.043 nm), two plane-convex lenses (focal lengths 300 and 50 mm), a pin-hole (diameter 1 mm), a polarizing-beam splitter cube and two *side-on* photomultiplier tubes. A lock-in amplifier operating on the chopper frequency is used to discriminate light scattering signals against flame luminosity and to reject out-of-band noise. Measurement volume (MV) (about 40×10^{-6} mm^3 at $\theta = 90°$) is spatially determined by the intersection of the laser beam incident on the flame axis and the detection cone (3.2° half-angle of

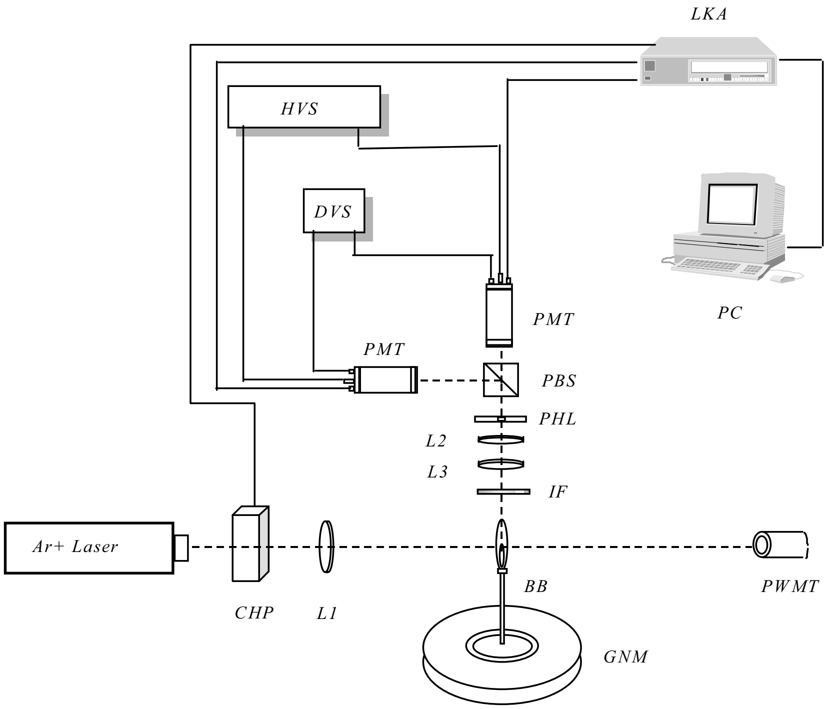

Fig. 1. Experimental set-up. Symbols: Ar^+=Argon Ion laser; CHP=mechanical chopper; L1=focusing lens (diameter 25.4 mm; focal length 50 mm); BB=Bunsen burner; GNM=goniometer; IF=interference filter; L2, L3=collecting lens (diameter 25.4 mm; focal length 300 mm); PHL=pin-hole (1 mm diameter); PBS=polarizing beam splitter; PMTs photo-multiplier tubes; PWMT=power meter; LKA=lock-in amplifier; HVS, DVS=high- and dual-voltage supply.

detection). Linear vertical polarization state of light radiation at laser output is changed in linear horizontal by passing the laser beam through a $\lambda/2$ retardation plate.

The information about the primary particle size, which is quite inessential to the results discussed in this work, is retrieved by SEM analysis of soot collected on quartz supports exposed directly inside the flame in correspondence of the measurement volume following a procedure detailed elsewhere [24].

Spherule size is recognized to be a function of both height above the burner (HAB) and of radial coordinate. All the measurements herewith reported are performed at radial coordinate zero (i.e., on flame axis) for different heights above the burner (HABs). The gyration radius R_g is obtained from the $I_{VV}(q)$ measurements corresponding to the Guiner regime ($q^2R_g^2 \ll 1$) where the equation $I_{VV}(q) \propto 1 - q^2R_g^2/3$ is valid for any cluster morphology [5], from the slope of $I_{VV}(0)/I_{VV}(q)$ vs. squared q. In analogous manner, the fractal dimension D_f is obtained within the power law $I_{VV}(q) \propto q^{-D_F}$ regime ($q^2R_g^2 \gg 1$) by a log–log plot of $I_{VV}(q)/I_{VV}(0)$ vs. q [5,20]. Guiner and power law approximations hold true for $I_{VV}(q)$ regardless of the particular cut-off function [21] and refractive index of soot. Experimental uncertainties of such methods are within 15% (SEM analysis) for primary particles, and 12%, 8% (light scattering) for radius of gyration and fractal dimension, respectively.

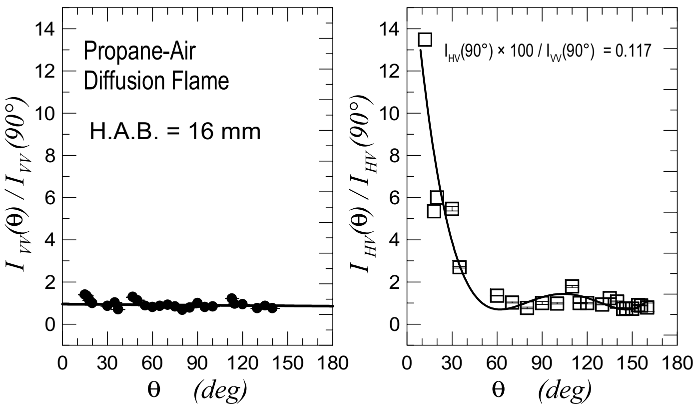

Fig. 2. Measured contributes of co-polarized (I_{VV}, left) and cross-polarized (I_{HV}, right) scattered light intensity vs. polar angle θ at 16 mm HAB and radial coordinate zero for a propane–air diffusion flame ($L_{flame} \sim 120$ mm, $v_{cold\text{-}gas} = 5.3$ cm/s, air inlet blocked).

3. Results and discussion

Fig. 2 shows an example of $I_{VV}(\theta)$ and $I_{HV}(\theta)$ measured at 16 mm HAB, at the centerline, in a propane air flame of apparent length 12 cm generated by the Bunsen burner. Measurements are corrected to account for the varying MV by multiplication times $\sin\theta$ and normalized at the 90° values of the angular pattern. SEM analysis of soot samples probed at the same HAB indicates that primary particles are about 16 ± 3 nm (one standard deviation σ experimental error on a set of 50 measurements). At 16 mm HAB they are still isolated or just start to agglomerate in clusters of a few spherules (dimers and trimers) with partial coalescence and formation of neck-like contacts.

The essential point to stress is that the angular pattern of I_{VV} is uniform with respect to θ, as expected for Rayleigh scatterers [15]. Similarly, the measured I_{HV} is characterized by a strong forward lobe at angles smaller than 40° and a wavy behavior at larger angles with a local minimum (80°) and maximum (110°) above a *plateau* (Fig. 2, right). The behavior observed for I_{HV} is in accord with previous results reported in the literature for acetylene/air and ethylene/air diffusion flames [25].

From a physical point of view, a small inelastically scattered contribution is expected to contribute to the depolarized elastically scattered light at smaller HABs as effect of the fluorescence radiation from larger polycyclic aromatic hydrocarbons (PAHs), for instance acenaphthylene [26], filtered within the detection band of the interference filter (1.043 nm FWHM centered at 514.5 nm). Nevertheless, angular patterns here reported are relative to HABs much larger of those corresponding to the first soot nucleation region [27], typically less than 5 mm in hydrocarbon diffusion lames [28].

On the other hand, the broadband and unpolarized fluorescence radiation is not depending on the observation angle. Thus, the physical reasoning that can be made to explain the higher angular sensitivity of the depolarized with respect to the co-polarized light to the onset of the agglomeration process, is that only the second-order contributions of the *multiple scattering* from the soot clusters

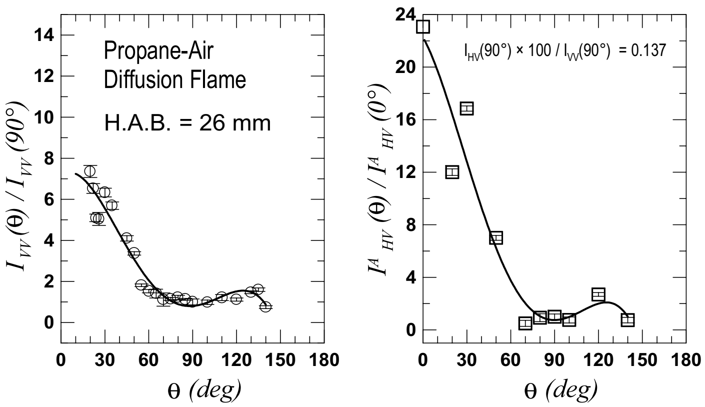

Fig. 3. Co-polarized (I_{VV}, at left) and cross-polarized (I_{HV}, right) scattered light intensity vs. polar angle θ at 26 mm HAB and radial coordinate zero for the same propane–air diffusion flame of Fig. 2.

(light induces electrical polarization at least inside two particles in the agglomerate before its arrival on detector) is sufficiently sensitive to the cluster structure as determined by the neighboring of each single primary particle [29]. Fig. 3 shows the angular pattern of I_{VV} and I_{HV} at HAB = 26 mm for the same propane–air diffusion flame. We observe from reduction of $I_{VV}(q)$ that fractal clusters with $D_f = 1.26$ and $R_g = 302$ nm do form. The primary size from SEM corresponding to 26 mm HAB is 22 ± 4 nm. The low value of D_f suggests that elongated assemblies of primary particles with branched chain morphology are structuring at this stage within the measurement volume. In correspondence of this situation, the measured vertical depolarization ratio $\rho_V \equiv I_{HV}/I_{VV}$, not shown here, exhibits a local minimum at angles about 80–90°. Absolute percent measurements of ρ_V are coherent with typical values of about 1% reported in the literature of flames [28,30]. However, these measurements of ρ_V here are considered with caution, owing to the fact that the measured ρ_V are too similar to the depolarization ratio expected for the unburnt cold-gas (propane) used as fuel [31]. Questions about depolarized light scattering from soot aggregates with more compacted and connected morphology has been addressed by further experiments conducted on an ethylene–air diffusion flame. Figs. 4 and 5 show the patterns of co-polarized scattered light I_{VV} measured at HABs of 10 and 30 mm. In this case, the same Bunsen burner is fueled (air-inlet blocked) by ethylene at cold-gas velocity 14.5 cm/s and the apparent flame length is 17 cm.

At the lower HAB (10 mm), mostly scarcely agglomerated particles are observed with a mean primary particles, as observed by SEM, equal to $D_P = 22 \pm 4$ nm. Some small aggregates of about ten particles are observed in the micrographs. The angular pattern of scattered I_{VV} light intensity are measured. The reduction of the experimental I_{VV} data vs. the momentum q yields to an inferred fractal dimension $D_f = 1.25$ and radius of gyration $R_g = 232$ nm. The measured vertical depolarization ratio ρ_V (Fig. 4, right) is found to be not sensitive (flat) with respect to the scattering angle and in magnitude lower than about 1%. At the higher HAB=30 mm, mature agglomerates are observed both in the SEM analysis and in the light scattering experiments. The reduction of the experimental data yielded a fractal dimension $D_f = 1.71$ and a radius of gyration $R_g = 163$ nm. The primary particle size from SEM analysis is on average 40 ± 7 nm. Thus, intra-cluster density is significantly larger with respect to the soot formed in propane–air flame. The observed $D_f = 1.71$ is very close to the typical

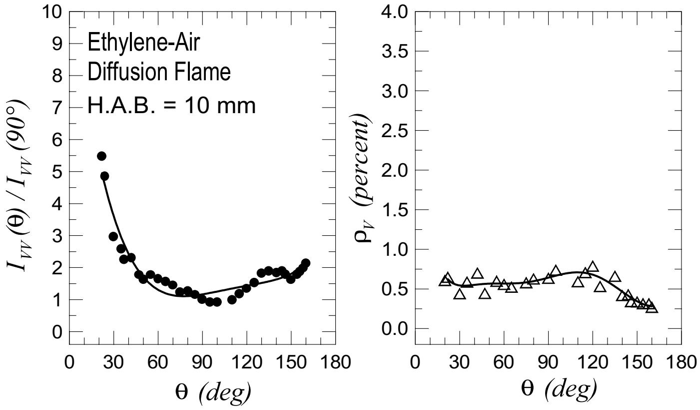

Fig. 4. Co-polarized (I_{VV}, at left) and vertical depolarization ratio $\rho_V = I_{HV}/I_{VV}$ (percent) vs. polar angle θ at 10 mm HAB and radial coordinate zero for a ethylene–air diffusion flame ($L_{flame}{\sim}170$ mm, $v_{cold\text{-}gas} = 14.5$ cm/s, air inlet blocked).

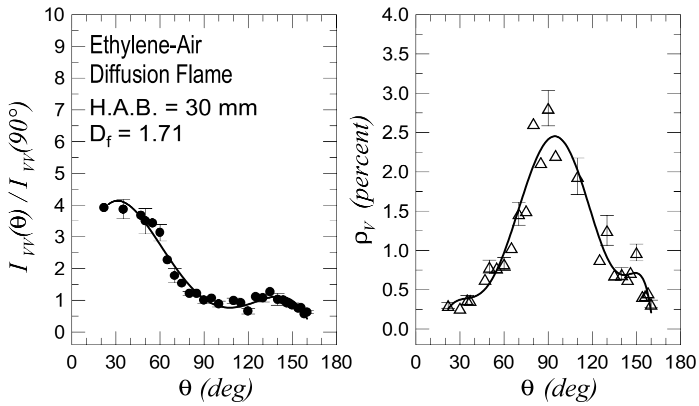

Fig. 5. Co-polarized (I_{VV}, at left) and vertical depolarization ratio $\rho_V = I_{HV}/I_{VV}$ (percent) vs. polar angle θ at 30 mm HAB and radial coordinate zero for the same ethylene–air diffusion flame of Fig. 4.

$D_f = 1.75$ value obtained by simulations of Diffusion-Limited Cluster–Cluster Aggregation, in which case clusters grow by mutual sticking as well as by monomer addition. The vertical depolarization ratio ρ_V (Fig. 5) shows, a broad local maximum between 80° and 120°.

From classical electromagnetic theory, we expect that for isotropic spheres $I_{HV} = I_{VH} = 0$, whereas for a single non-spherical particle $I_{HV} = I_{VH}$, that is known as the *reciprocity theorem* (for a review see [32]). Nevertheless, different authors [2,32] reported numerical results that predict the non-validity of the reciprocity relations for soot aggregates.

We questioned about which HAB effectively corresponds to the onset of the early agglomeration process. In principle, the information could be obtained by the measurements of the angular pattern, if one consider that for non-agglomerated Rayleigh sphere the I_{VV} should be not depending on θ. Nevertheless, this point is complicated by the fact that in the near forward scattering region a

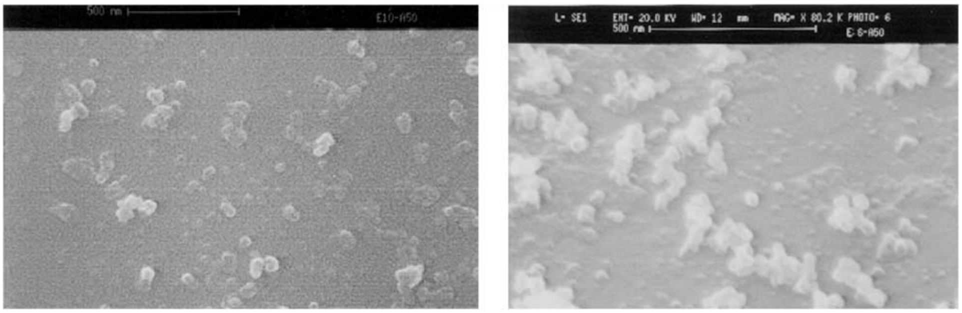

Fig. 6. SEM micrographs of the ethylene soot collected by thermophoretic sampling on the flame axis at 10 (left), and 16 mm (right) height-above-burner (HAB) for the same ethylene–air diffusion flame of Figs. 4 and 5.

considerably larger measurement volume MV is obtained. Thus, information drawn from measurements could be influenced by the larger particle polydispersion and/or by the different physical properties of the soot particles with respect to those on the flame axis. Ideally, we would recognize some information from measurements at 90° where a pin-sharp MV can be used.

The first approach to the above question is to investigate by thermophoretic sampling and SEM analysis, albeit the introduction of a quartz disc for probing is recognized to somewhat influence and introduces bias to the detection of larger particles (single or aggregates of a few sub-units). In particular, the thermophoretic force could be size-dependent, thus dropping down for smaller particles. The net effect would be to reveal a prevalence of the larger particles. In Fig. 6, we report the images of two samples taken at 10 and 16 mm HAB as observed by SEM. From the latter (Fig. 6, right) it is clear that particles effectively stick to each other at this stage. Also at 10 mm (Fig. 6, left) some clusters of a few primary particles are noticed. Thus, it is concluded from this preliminary SEM study that the onset of agglomeration does occur at heights of about 10 mm, or probably lower (on the basis of an eventual size dependence of thermophoretic probing).

We next turn to the measurements of the depolarized light. The ratio of measurements of I_{HV} (incident vertical polarized light) to I_{VH} (incident horizontal polarized light) is shown in Fig. 7 against the scattering angle. The major evidence here is that, in contrast with the theoretical prediction of the reciprocity theorem [32], the measured ratio I_{HV} to I_{VH} is other than unity for aggregated soot nanoparticles, except at forward ($\sim 15°$) and backward ($\sim 160°$) scattering angles.

In particular, the measured ratio I_{HV} to I_{VH} results about unity, within the experimental error, for non-agglomerated Rayleigh particles (HAB < 8 mm) and exhibits a strong peak at about 100–130° in the regime where branched linear chains ($D_{\mathrm{f}} \approx 1.3$) form within the flame (HAB > 10 mm). Comparing our experimental data with numerical results from literature, we find qualitative agreement with Ku and Shim [2] who reported numerical calculation of $I_{\mathrm{HV}}/I_{\mathrm{VH}}$ by ICP approach assuming chain soot aggregates with refractive index $m = 1.7 + 0.7i$ and primary size parameter $x_{\mathrm{P}} = \pi a_{\mathrm{P}}/\lambda = 0.1$. In contrast to our measurements, their numerical calculations predict smaller $I_{\mathrm{HV}}/I_{\mathrm{VH}}$ values at about 90°, thus, underestimating our experimental results. This is probably the effect of both the arbitrary assumption of the refractive index value used for computations [2] and, also, of the eventual polydispersion of both primary particle size and agglomerate shapes. The experimental results presented here support the feasibility of a simple experimental procedure to discriminate in

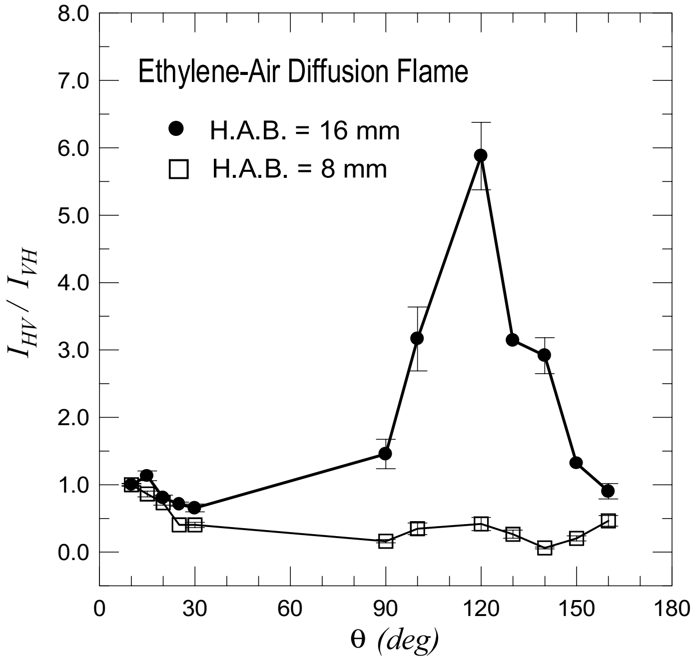

Fig. 7. Ratio of the cross-polarized scattered light intensities for the two possible (vertical and horizontal) linear polarization states of incident light as function of polar angle. Same flame of Figs. 4–6.

situ between the two cases of isolated and agglomerated soot nanosize particles starting from the measurements of I_{HV} to I_{VH} in a very narrow range of scattering angles at about 120°.

4. Conclusions

In this paper, we have reported, for the first time to our knowledge, experimental evidence that can be used to detect the onset of agglomeration process in flame generated soot particles. Major results can be summarized as following: (a) in the case of smaller soot agglomerates with open structured branched chain appearance ($D_f \sim 1.3$), such as in the case of the propane–air flame at lower HABs, the vertical depolarization ratio ρ_V is characterized by essentially a flat behaviour with very small values (< 0.01); (b) when more structured fractal soot is generated ($D_f \sim 1.7$), the angular pattern of ρ_V is characterized by a broad maximum at θ in the range 80–120°; (c) more sensitivity to the onset of the agglomeration process by depolarized light component I_{HV} is observed with respect to the co-polarized scattering term I_{VV}. In particular, a sharp maximum at about 120° is measured for the ratio I_{HV}/I_{VH} at the very early stages of the agglomeration process.

This experimental evidence demonstrates the feasibility of a very easy laboratory method to sense in situ with high spatial resolution (~ppm of mm^3) the unknown eventuality of agglomeration within a nanoparticle system. The outlined conclusions are expected to be very useful in the gas-phase combustion synthesis of weakly absorbing inorganic nanoparticles [33] for possible application in high-temperature superconductors, electronic substrates and catalysts when agglomeration must be avoided to accurate control of particle morphology.

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