Granulometric Composition of Settled Aerosol Material and Ratio of Phenolic Compounds in Different-Age Needles

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Abstract—We present the results from laser granulometry of a nanosized fraction of aerosol substance and from UV spectrometry of water washouts from different-age needles in four plant species. It is estimated how the hydrothermal conditions influence the time variations in morphometric indices of needles and contamination of their surfaces by phenolic compounds in the composition of secondary organic aerosols. We discuss how secondary organic aerosols are generated on the surfaces of different-age needles during efflorescence of phenolic compounds and their subsequent entry into the forest canopy during turbulent separation of the boundary layer from the leaf surface.

INTRODUCTION

Leaves of coniferous trees are known to play an important regulating role in the functioning of the plant organism [1]. It is noted that any deviations in morphometric indices of needles have an unavoidable effect on the characteristics of the assimilating surface and, hence, entail changes in the productivity and viability of the plant organism during its development [2]. At the same time, observations [3] indicate that needles actively grow in size only during the first year. Therefore, the surface area of the leaf can be thought to change very little until the needle falls. Hence, with respect to aerosol fallout events, the leaf surface of coniferous plants can be considered as a natural tablet computer, reflecting specific features of passive aerosol accumulation throughout the needle lifetime. Considering that this period is from 3–6 to 12–17 years for different tree species, different-age needles can be an informative testing entity in estimating aerosol pollution of the surface atmosphere.

Granulometric composition of aerosol material can differ in plants whose needles have different lifetimes. The pine needles live for 3–6 years (Scotch pine is very sensitive to atmospheric pollution, such as the yellow Chernobyl forest). The spruce needle lifetime varies from 7 to 14 years (spruce well assimilates under urban conditions, in contrast to pine), and fir needles live for up to seven years. Depending on exposure time, needles of different ages are thought to differ by granulometric composition of aerosols. Therefore, the washouts from needles of different tree species make it possible to compare which of the conifers are more informative for monitoring aerotechnogenic pollution.

In this regard, the purpose of this paper is to study the granulometric composition of a nanodisperse fraction of aerosol material by the method of dynamic light scattering in water washouts from needles in four plant species, and to compare the relative contents of phenol compounds as functions of morphometric characteristics and different needle lifetimes.

TECHNIQUE

Studies were carried out at test sites at the end of the growing season (the last decade of September 2020). The sites were arranged on the territory of the Botanic Garden in Pitirim Sorokin Syktyvkar State University, located 4 km west of Syktyvkar in the suburban green zone. The studies concentrated on four types of coniferous trees: 18–25-year-old Siberian pine (*Pinus sibirica* Du Tour) and Scotch pine (*Pinus sylvestris* L.), 10-year-old undergrowth of European fir (*Abies alba* Mill.), and 30–40-year-old Siberian spruce (*Picea obovata* Lebed.). Needle samples were

selected with respect to age fractions for morphometry and taking washouts using second order branches in the lower third of the crown in each tree species within eastern bearings (in azimuthal sector between 45 and 135°).

Dynamic Light Scattering Technique

The dynamic light scattering (DLS) technique, related to nondestructive methods of research, has become widespread recently among the possible integrated techniques for studying nanoparticles in disperse media. The physical essence of the technique is that time fluctuations in the intensity of a laser beam scattered in a disperse medium are recorded. Owing to Brownian particle motion, fluctuations in local particle concentrations and the associated local changes in optical density occur in the disperse medium, as well as the related variations in refractive indices. The latter is because light is partially scattered as the laser beam passes through local particle concentrations. Numeric parameters of light scattering are found to depend on particle sizes, the intensity of their diffusion, and the viscosity of the liquid [4]. The technique does not require a preliminary calibration, and it is equally efficient both for low concentrations of particles, and in the presence of their aggregates. The measured particle size (diameter) varies from 0.5 nm to a few microns. The technique is characterized by the low cost of measurements, small error, and rapidity.

Preparation of Washouts from Needles for Granulometric Analysis and UV Spectroscopy

We placed needle specimens in glass beakers, 30 pieces in each age fraction, and added deionized water (50 mL). Then, the beakers were transferred to an ultrasonic bath Sapfir UZV-5.7 (working frequency 35 kHz, and generator power 150 W), in which the specimens were exposed to ultrasound for 5 min. A ZetaSizer Nano ZS laser analyzer (Malvern Panalytical, Great Britain) was used to determine nanoparticle sizes in samples. Particles from 1 to 10000 nm were measured. The average volumetric particle diameter (D_{aver}, nm) was measured six times for each sample. In each measurement, the optimal accumulation time of the time autocorrelation function of scattered radiation intensity was automatically determined by the instrument software; they were then averaged. The volume content of nanoparticle fractions in the samples was integrally calculated from the ratio (%) of the areas of the figures circumscribing these particle size distributions in linear coordinates. The Solar PB2201 ("Spectroscopy, Optics, and Lasers—Advanced Developments" Ltd., Belarus) spectrophotometer was used to carry out UV spectroscopy of water washouts from needles.

RESULTS AND DISCUSSION

Frequency Distribution of Particles in Aerosol Material over Sizes in Water Washouts from Longleaf Plants (Siberian and Scotch pines)

Comparison of granulometric compositions of aerosol material with respect to age fractions in water washouts from longleaf plants showed that the particle diameter range in five age fractions of Siberian pine is from 70 to 2000 nm. The particle distribution is predominantly bimodal. It should be noted that D_{aver} varies in the range 135−357 nm, with no time trend found in growing particles (Fig. 1, A-*1*−A-*5*). Comparison of granulometric compositions of aerosol material with respect to age fraction in water washouts from Scotch pine showed that the size range of particles in three time fractions is 60−1500 nm. The fraction of fine (60−150 nm) particles is strongly predominant in the relationship of size fractions. At the same time, D_{aver} varies in the range 161−273 nm, with no direct relationship found between growth in the average particle diameter in aerosol material and an increase in the leaf lifetime (Fig. 1, B-*1*−B-*3*).

Thus, the granulometric composition of disperse aerosol material in longleaf plants is characterized by a predominantly bimodal distribution. The fine (particle sizes of 60−200 nm) fraction is strongly predominant in the ratio of fractions; and the analysis of the total variability range of the optical density in water washouts from Siberian and Scotch pines revealed significant differences, which suggests that the needles in the latter are more heavily contaminated.

Frequency Distribution of Particles in Aerosol Material over Sizes in Water Washouts from Shortleaf Plants (European fir and Siberian pine)

Comparison of granulometric compositions of aerosol material with respect to age fraction in water washouts from shortleaf plants showed that the particle diameters in four age fractions of the needles of European fir vary in the range from 50 to 1800 nm. The particle distribution is predominantly bimodal. The fine fraction of particles with sizes of 50−100 nm is strongly predominant in the relationship of size fractions. At the same time, D_{aver} varies in the range 78−199 nm; we can note that, as the needles of the European fir are aging, particles of aerosol material grow in average diameter (Fig. 1, C-*1*−C-*4*). Comparison of granulometric compositions of aerosol substance with respect to age fractions in water washouts from Siberian pine showed that particles in four age fractions are within the size range 50−1200 nm. The distribution is predominantly bimodal with a large variance. The fine (50−140 nm) particle fraction are very strongly predominant in the relationship of size fractions. The D_{aver} value tends to decrease as needles of Siberian pine are aging.
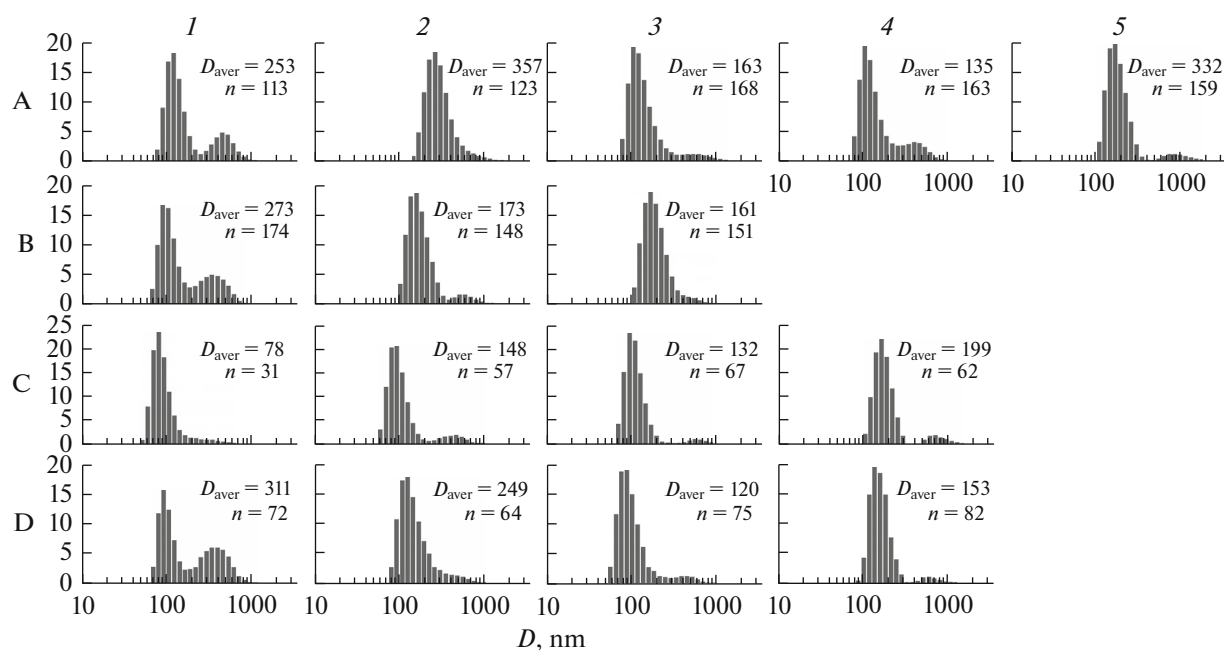
Fig. 1. Granulometric composition of aerosol substance in water washouts from different-age needles ((*1−5*) indicate needle age in years): A is for Siberian pine (*Pinus sibirica* Du Tour); B for Scotch pine (*Pinus sylvestris* L.); C for European fir (*Abies alba* Mill.); D for Siberian spruce (*Picea obovata* Lebed.); and *n* is the number of measured needles in sample gone to calculate the leaf surface in each age fraction.

Thus, a common feature for all needle specimens is that the granulometric composition of different-age fractions of needles is characterized by a bimodal distribution of nanoparticles; and small particles, whose fraction reaches 91−96%, are predominant in the size relationship. Hence, this leads us to speculate that large particles on the needle surface can represent solid aggregates of smaller particles. These primary particles in such clusters are bound by intermolecular interaction forces. The aggregation of small particles is associated with their enlargement (size and mass growth of aggregates), and reflected in their decreased numbers in the volumes of water washouts from needles. In other words, the aggregation process of small particles on the needle surface leads to the formation of the secondary large particles.

UV Spectroscopy of Water Washouts form Different-Age Needles

Figure 2 shows the absorption spectra of water washouts from needles of different tree species. It is clearly seen that water washouts from all specimens absorb quite strongly radiation in the UV region, with absorption intensity gradually weakening in going into the longer-wavelength region. This behavior is characteristic in the presence of scattering disperse particles. At the same time, absorption zones, characteristic for each tree species type, exist in the spectra. For instance, for different-age spruce needles there are three absorption intervals in the wavelength range 250−400 nm, with the maxima at 274, 306, and 322 nm (Fig. 2, D-I). For fir needles there is the only explicit absorption zone with the maximum near 270 nm (Fig. 2, C-I). In the case of the needles of Scotch pine, the spectra have three maxima: 282, 324, and 370 nm (Fig. 2, B-I). In the spectrum of washouts from needles of Siberian pine there is the only flat peak in the range 260−300 nm (Fig. 2, A-I). These segments can indicate that the washout samples comprise phenolic compounds (flavones, flavonoids, phenylpropenes, quinones), comprised by needles [5, 6].

It can be seen that the content of phenolic compounds increases with needle age, consistent with an earlier finding [7]. This specific feature is considered to be because of reduced wetting of cells during their aging and lignification. But then, in this context the anomalous increase in the content of phenolic compounds in two-year-old needles of Scotch pine (Fig. 2, B-I) and their rapid decrease in three-year-old needles requires a different explanation.

UV Spectroscopy of Water Washouts from Different-Age Needles after their Centrifugation

Experiments with water washouts from needles were continued after a comparative analysis of UV spectra of water washouts from different-age needles in four plant types. Figures 2, A-II−D-II show how the optical density of water washouts from needles changed after one month of settling and a subsequent centrifugation. After subtracting the spectra: 1) before
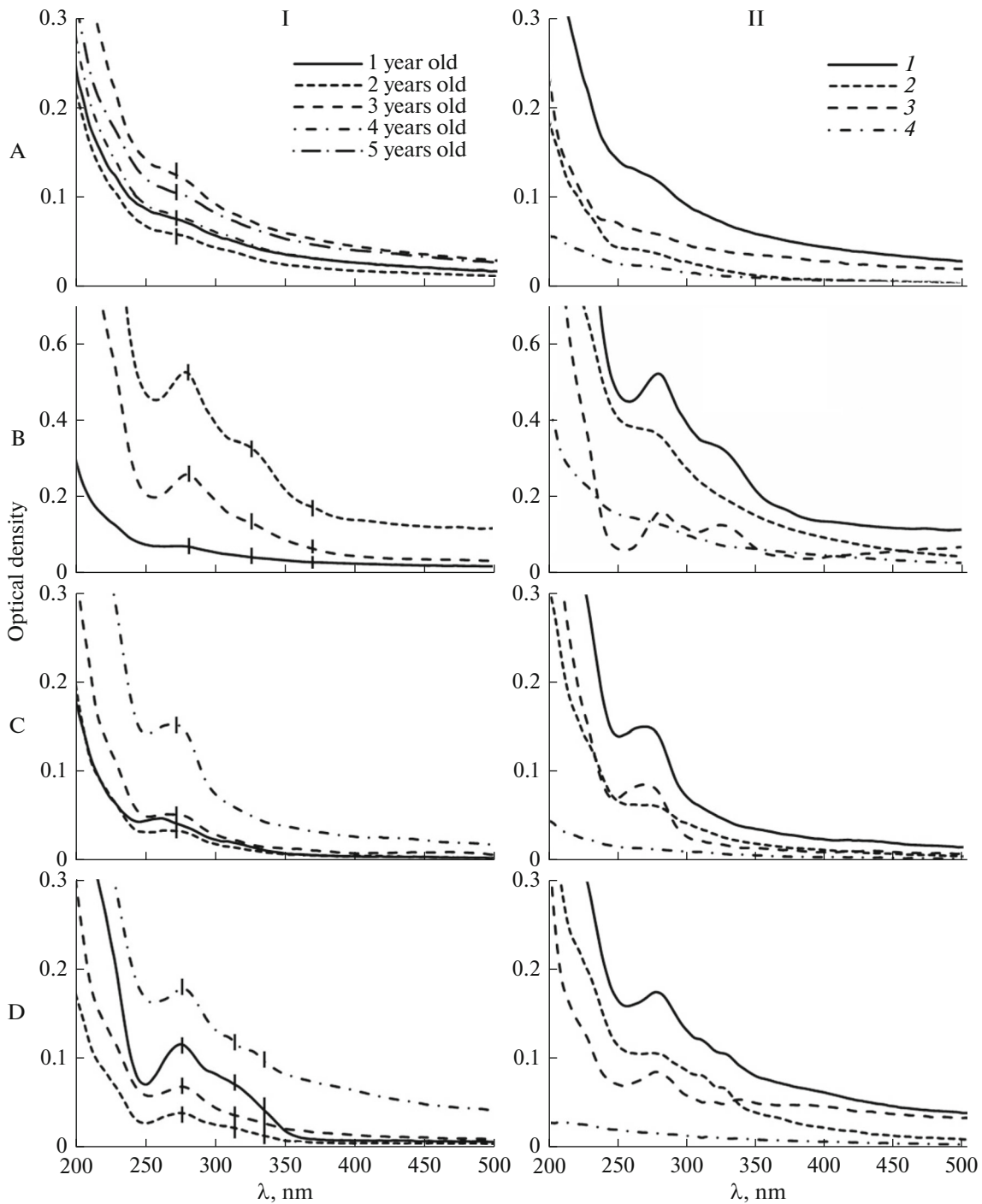
Fig. 2. (I) UV absorption spectra of water washouts from different-age needle fractions: A is for Siberian pine, B for Scotch pine, C for European fir, D for Siberian spruce; (II) UV absorption spectra of water washouts in a single age fraction: A is for (3 years old) Siberian pine, B for (2 years old) Scotch pine, C for (4 years old) European fir, D for (4 years old) Siberian spruce; shown are the initial spectrum (curve *1*), spectrum obtained after 1 month of settling (curve *2*), spectrum characterizing large deposited particles, obtained by subtracting optical densities before and after settling (curve *3*), and spectrum characterizing small deposited particles, obtained by subtracting optical densities before and after centrifugation (curve *4*).

and after settling (Figs. 2, A-II(*3*)−D-II(*3*)); 2) before and after centrifugation (A-II(*4*)−D-II(*4*)), we obtained the absorption spectra characterizing deposited particles. The above-indicated reduction of the optical density indicates that coarse particle fractions settled in the first case, and fine particle fractions in the second case. In contrast to large particles, small particles in their UV spectra have no absorption bands, characteristic for needles and observed in initial spectra (A-II(*1*)−D-II(*1*)).

These findings can indicate that emissions of phenolic compounds (products of intra-cellular metabolism) form relatively solid aggregates, i.e., the secondary organic aerosols, on the needle surface. This possibility for conversion of gas-phase phenol compounds was shown in many works (e.g., [8, 9]). These primary phenolic compounds can be considered as something like precursors of the secondary organic aerosol, which can be detected and characterized using UV spectrometry, this latter because phenolic compounds have characteristic absorption spectra in certain regions of the UV spectrum [10].

Specific Features of Formation of Secondary Organic Aerosols in the Coniferous Forest Canopy

The formation of the secondary aerosols with the participation of phenolic compounds is thought to be associated predominantly with their condensing on the surface of nanodisperse aerosol material [11]. The presence of these compounds was revealed in experiments on determining the optical density of water washouts from different-age needles after their centrifugation. However, there can also be some other way they form in the coniferous forest canopy, namely, efflorescence. This is a surface phenomenon, associated with migration of phenolic compounds in the composition of transpiration moisture and its evaporation with a subsequent crystal formation under the influence of UV radiation and free-radical oxidation on the needle surface.

Generation of Secondary Organic Aerosols and Their Supply to the Forest Canopy

Moisture transpiration is often accompanied by removal of the gas-phase products of cellular metabolism, which in the environment experience certain chemical conversions, thus changing their chemical activity and, often, their phase state, transiting from gas to liquid state, and from liquid to solid state. Visually, this is often found to look like salt powders (salt effusion) on the leaf surface (in the English-language literature, this process is designated by phrase *salt efflorescence*).

An example of efflorescence can be the description of how salt efflorescence forms on the grass surface under arid conditions on the territory of Mpumalanga highveld of the Republic of South Africa [12]. The mineral composition of salt efflorescence was studied using X-ray diffraction, differential thermal analysis, scanning electron microscopy, and chemical analysis. As a result, it was revealed that the mineralogical paragenesis of salt efflorescence was formed from thenardite, bloedite, gypsum, and kieserite. The authors of work [12] conclude that all these minerals were formed during the six dry winter months. They also presented the calculations showing that the approximate rate of thenardite (most ubiquitous mineral in the studied mineral paragenesis of salt efflorescence) deposition can be up to $11-15$ g/m^2 [12].

It should be noted that all these minerals represent readily water soluble minerals in the sulfate class. They are deposited from Na_2SO_4-rich solutions as associated minerals. Under the conditions of relatively low air temperatures (+20 to +30°C) they precipitate from solutions as crystalline hydrates. However, if we take into consideration the data on mineralogical composition [12], which indicate the presence of kieserite, the efflorescence would be detected in the dry period not only on plants, but also on the soil surface, which was not even mentioned in [12]. Without doubts in reliability of the data presented by investigators in the mineralogical composition of salt efflorescence, taken from the surface of plants, we can speculate that kieserite appears in their composition due to deposition of the mineral on the grass surface in the form of dry aerosols.

In this regard, we should give the following clarifications. Kieserite is a mineral, a hydrated magnesium sulfate. It comprises: 29% MgO, 58% SO_3, and 13% H_2O. In fog, which is mentioned in paper [12] as one of the necessary conditions for occurrence of salt efflorescence on plant surfaces, this mineral could never be formed because kieserite in moist air is covered by a film of epsomite, i.e., a water-soluble mineral (16.3% MgO, 32.5% SO_3, and 51.2% H_2O), also being a member of the sulfate class. Under the conditions described above, kieserite could only form by metamorphism of epsomite via its dehydration under the conditions of low relative humidity and high daytime temperatures.

At the same time, the development of convection and turbulence in the surface air layer is known to be determined by the gradient and diurnal variations of temperature in the active layer ($0.01-0.5$ m), which are $1.5-2.0$ times larger than those recorded at an altitude of 2 m. As shown in [13–15], the vortical entrainment and outflow of aerosol from the soil surface can be initiated under these conditions. Therefore, epsomite could by supplied to the surface air layer after being entrained by airflows from the soil surface as a result of thermophoresis with a subsequent conversion to kieserite in the flow of hot air and its dry deposition as a result of thermal precipitation on the "cold" surface of desert plants.

The thermophoresis (removal of aerosol particles from a heated surface) and thermal precipitation (deposition of aerosol particles on colder parts of the surface) are known to be specific aerosol properties [16]. It is noteworthy that air is transparent to solar radiation; therefore, air temperature depends on the heating of aerosol particles, and this latter, in turn, depends on the intensity of solar radiation. Hence, the greater the number of particles in the boundary layer, the larger the layer temperature, and the stronger the molecular mixing: after being heated, particles transfer
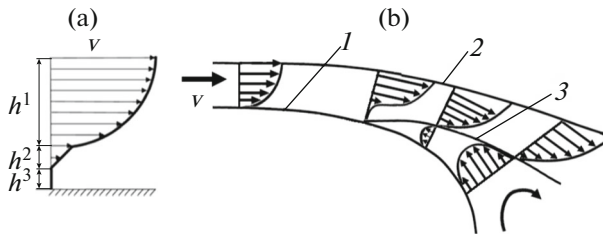
Fig. 3. (a) Boundary layer and its separation: h^1 is the turbulent layer, h^2 is the laminar layer, and h^3 is the buffer sublayer; (b) surface of the body (curve *1*); boundary of the boundary layer (curve *2*); and boundary of the boundary-layer separation (curve *3*).

a part of their kinetic energy to air molecules in the boundary layer.

Activation of secondary organic aerosols and their subsequent supply to the plant canopy begins in the *boundary layer*. The boundary layer can be *turbulent* or *laminar*, depending on how the speed *v* of airflow is distributed [17]. The turbulent boundary layer is characterized by nonlinear speed distribution (Fig. 3a). In the laminar boundary layer, the speed of the airflow varies linearly: from zero on the solid surface to *v* at the boundary between the layer and airflow. Because of surface roughness, a buffer sublayer with zero-speed airflow exists between the laminar flow and the solid surface. As the speed of the airflow increases, the laminar layer becomes turbulent and much thinner.

Reverse flow and boundary-layer separation. A specific feature of the boundary layer is that it admits, in certain circumstances, the occurrence of reverse flow in the immediate vicinity of the surface. This property is associated with the *separation of the boundary layer* from the surface and the occurrence of a vortical zone in the region of variation in the pressure gradient. The necessary condition for the boundary layer separation is the increasing pressure in the direction of the airflow. As the airflow circumvents a curvilinear or roughed surface, the pressure and speeds at different points on the surface will be diverse (Fig. 3). The pressure grows along the trajectory of the airflow, and the speed of the airflow down the boundary layer profile decreases; therefore, a reverse gas motion occurs in response to the pressure difference. It is noteworthy that the airflow continues to move in the longitudinal direction over the surface, but deflects somewhat away from the surface due to backward gas flow. Occurrence of a reverse flow leads to a considerable thickening of the boundary layer, loss of its stability, and airflow separation from the surface [18]. The separation process is accompanied by the formation of a vortex zone and a subsequent transition to turbulence. It is assumed that the inflow of secondary organic aerosols to the forest canopy is associated with a similar process of perturbing the stability of the boundary layer.

Conditions for turbulence formation in forest canopy. The development of turbulence in the surface air layer is determined by the gradient and diurnal behavior of the temperature in the active layer. In the forest, crowns begin to play a role of an active surface [19]. During clear-sky weather, the surfaces of crowns strongly heat due to the influx of solar radiation. It is noteworthy that, due to nonuniform distribution of solar radiation and temperature in the forest canopy [20], the heating degree of the forest plants will vary at different forest stand levels. This temperature distribution favors the development of convection and turbulence in the forest canopy and, thereby, promotes the activation of turbulence in tree crowns.

CONCLUSIONS

We present the comparative studies of granulometric composition of nanodisperse fractions of aerosol material and the relationship of phenolic compounds and water washouts from different-age needles in four plant types. For all needle specimens we found common properties: the granulometric composition of different-age needle fractions is characterized by a bimodal distribution of nanoparticles; and the size relationship is dominated by small particles accounting for as much as 91−96%. Therefore, we can hypothesize that large particles on needle surface can represent robust aggregates of smaller particles. These primary particles in such clusters are bound by the intermolecular interaction forces. The aggregation of small particles is accompanied by their enlargement, reflected in their decreased number in the volume of water washouts from needles.

UV spectrometry of water washouts from different-age needles, both after their settling, and after their centrifugation, revealed a decrease in the optical density, indicating settling of large particles in the first case, and small particles in the second case. It is noteworthy that the UV spectra for small particles, in contrast to those for large ones, have no features characteristic of absorption bands of phenolic compounds observed in initial spectra.

It is postulated that phenolic compounds are found to form, on the needle surface, relatively robust aggregates with aerosol material, which can be considered as something like precursors of the secondary organic aerosols. Their activation begins in the boundary layer; and their subsequent supply to the plant canopy owes to the boundary layer the property to admit, in certain circumstances, the occurrence of reverse flow and separation of the boundary layer from the surface. The separation process is accompanied by vortex formation. This is presumed to be initiated by turbulence developed in the surface air layer, and to be determined by the gradient and diurnal behavior of the temperature in the active layer. It is noteworthy that, owing to the nonuniform distribution of solar radiation, the degree of heating of the forest plants will vary

at different forest stand levels, favoring the development of convection and turbulence in the forest canopy.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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