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Particulate Characterization of Herbal Extracts for its Standardization and Quality Control

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We have examined herbal extracts of *Hippophaë rhamnoides L.* (sea buckthorn), *Arnica montana L.* (arnica), *Malva sylvestris L.* (mallow). Sea buckthorn extract is an antiviral agent, arnica extract – anti-inflammatory agent and mallow extract – expectorant. MasterSizer 2000 and ZetaSizer Nano ZS (Malvern Instruments) were applied. Size spectra of herbal extracts were too irregular and specific for each sample. Dried extract of *Arnica foliosa Nutt.* characterize by prevalence of 1 μm and 3 μm particles by number and of 40 μm and 140 μm particles by volume. After dissolving only 1 μm and 3 μm particles were detected. Dried extract of *Malva sylvestris L.* characterize by prevalence of 1 μm particles by number but its volume distribution was too irregular (from 10 μm to 800 μm). In samples of sea buckthorn extract we have detected three groups of particles with size 50 μm, 150 μm and 200 μm. Inclusion of sea buckthorn extract in plaster mass caused disappearing of 50 μm and 150 μm particles with prevalence only of 200 μm particles. Polydispersive system changes into monodispersive due to inclusion of dried extract in plaster mass. It is a basic precondition of uniform distribution of dried extract particles which depends on biological activity and very important in application dosage forms technology.

1. Introduction

For dispersive analysis of pharmaceutical particulate systems such as powders and suspensions United States Pharmacopoeia (USP) and European Pharmacopoeia (EPh) recommend to use laser diffraction technique. Sedimentation analysis and microscopy have been using before are inappropriate to particulate characterization. Microscopy cannot give us total characteristics of particulate system cause of analyzing just a small part of the sample (Syroeshkin, 2005). Sedimentation analysis is low informative and takes a lot of time. There are a lot of other methods of dispersive analysis such as X-ray scattering, FT-IR spectroscopy and others. But making use of low-angle laser light scattering (LALLS) and dynamic light scattering (DLS) for analysis of pharmaceuticals is very informative and perspective. Total information on particulate system such as numerical and volume particle size distribution, specific surface area etc. are the main results of LALLS and DLS (Frisvad, 2007).

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1.1 Characters, action and use of herbal extracts

Sea buckthorn dried extract (also called “hyporamin”) is an original russian preparation. Hyporamin is obtaining from sea buckthorn leaf. It is a polyphenolic complex of gallo ellagitannins with the prevalence of hydrolysable tannins. It is a pale yellow amorphous powder, hygroscopic. Hyporamin is a powerful antiviral agent. It is especially effective against viruses of herpes and flu types A and B. Arnica dried extract is a yellow-brownish amorphous powder, hygroscopic, specific odoured. It is obtaining from arnica flower. Basic compounds are sesquiterpenes, phenyl carbonic acids, tannins, hydrocarbons. Arnica extract is a perfect anti-inflammatory agent. Mallow extract is a pale brown amorphous powder, hygroscopic, specific odoured. Mallow herb and root are main sources for extract obtaining. Main compounds are polysaccharides and tannins. It is a good expectorant.

1.2 Perspectives of LALLS and DLS

Particle size distribution of heterogeneous systems was studied in various fields of science from colloidal chemistry to cytology and pharmaceutical chemistry. It is very important characteristic of particulate systems. LALLS and DLS techniques have been introduced into many types of industry such as pharmaceutical, chemical, food etc. The major advantage of these techs is a possibility to find out a correlation between the properties of particulate system and its particle size distribution (Syroeshkin et al., 2005). Existence of such dependence has been determined by features of these techniques. Statistical morphometric estimation is carrying out for the whole sample, i.e. analyzing all particles. Moreover, the information on numerical and volume particle size distributions allows revealing fractions with a small number of large particles.

2. Materials and Methods

In this work scanning electron microscopy (SEM), low-angle laser light scattering (LALLS) and dynamic light scattering (DLS) were applied. Scanning electron microscope JOEL JSM-6490LV, particle sizers MasterSizer 2000 and ZetaSizer Nano ZS (Malvern Instruments) were applied as apparatus. MasterSizer 2000 measure range lies from 20 nm to 2000 μm, ZetaSizer Nano ZS measure range lies from 0.4 nm to 6 μm. For dispersive analysis follow sample preparations have been used:

1) Solutions of hyporamin. Solvent - mixture of distilled water and polyethylene glycol (4:1). We have compared this solution with the same solution of hydroxypropyl methyl cellulose plaster mass with hyporamin. Background - blank mixture and plaster mass solution.

2) Sample of dried mallow extract. Background - air.

3) Samples of dried arnica extract and its solution in mixture of distilled water and polyethylene glycol (4:1). Background – air and blank solution.

3. Results and Discussion

3.1 SEM pictures

Figure 1 shows the results of scanning electron microscopy in different zoom. All samples have been characterized by individual form and size of particles. Most of

hyporamin particles have a form of spheres, spatial polyhedrons and plates. Particle size range has been found from 2.5 µm to 45 µm. Form of mallow extract particles is different too. There are plates and parallelepipedons with the smooth surface. Particle size range has been found from 5 µm to 100 µm. Arnica extract particles are presented as thin plates and globules with the size range from 5 µm to 60 µm.

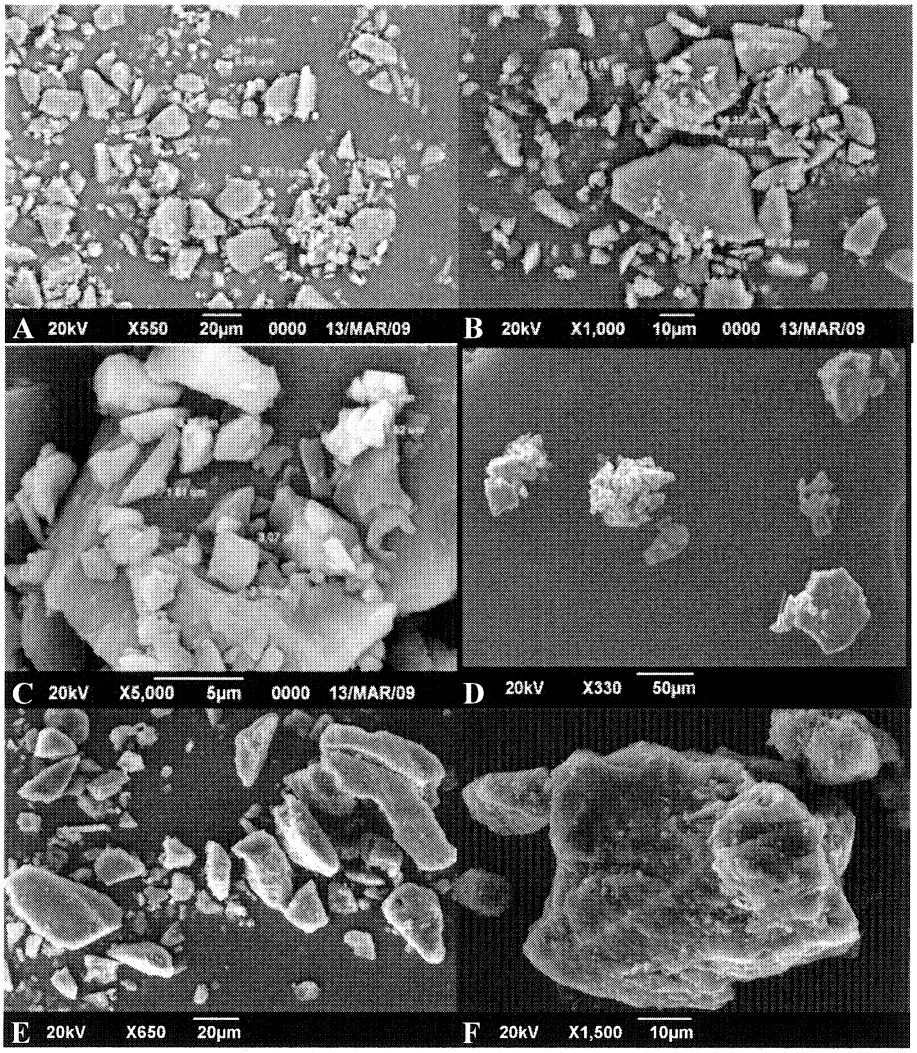

Figure 1: SEM pictures of dried herbal extracts. A,B,C – sample of hyporamin, D – sample of arnica extract, E,F – sample of mallow extract.

3.2 Size spectra

Figure 2 shows the size spectra of volume and numerical distribution of particles in herbal extracts samples. All size spectra are individual for each sample.

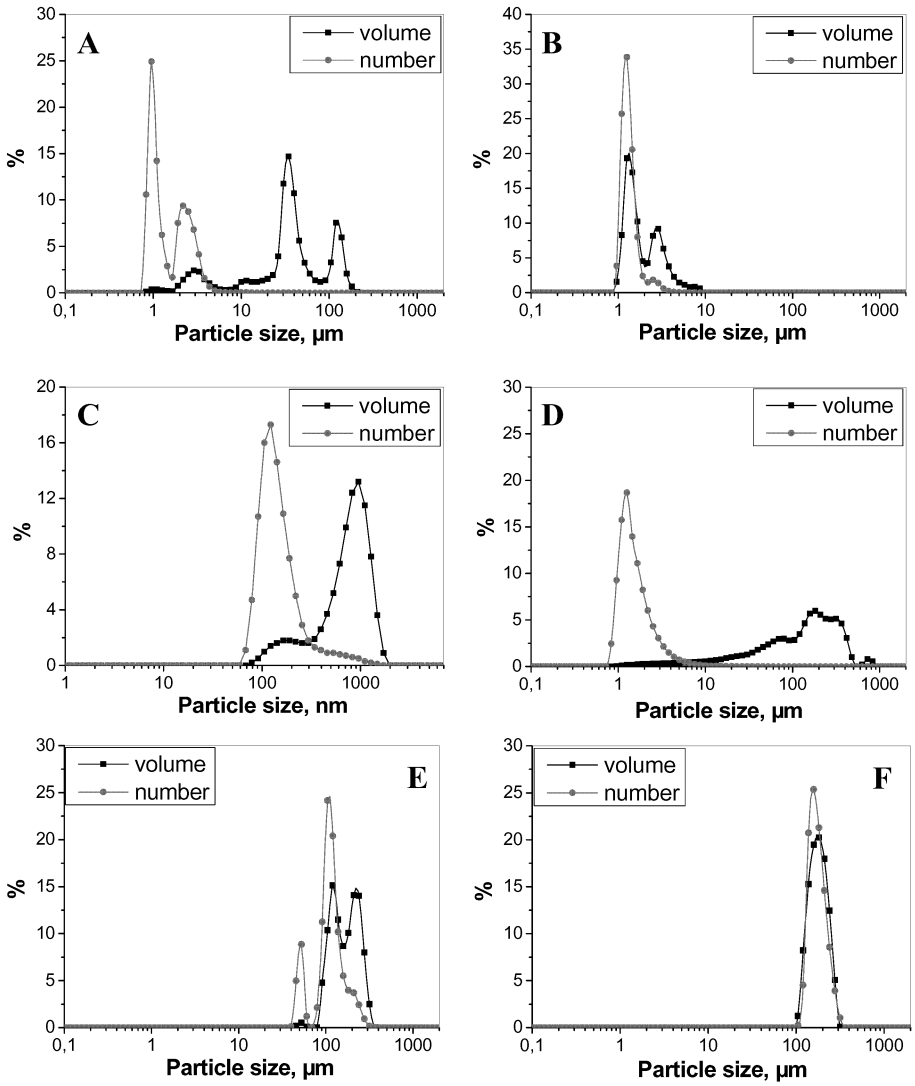

Figure 2: Size spectra of herbal extracts. A – sample of arnica extract before dissolution, B - sample of arnica extract after dissolution in microscale range, C - sample of arnica extract after dissolution in nanoscale range, D – sample of mallow extract, E – sample of dissolved hyporamin, F - sample of dissolved hyporamin plaster mass.

We have found five size groups of particles in samples of dried arnica extract with mean diameter 1, 3, 10, 40 and 150 µm. 1 µm and 3 µm particles prevail by number but characterize by low volume contents. Share of 10 µm particles is low in both distributions. Larger particles such as 40 µm and 150 µm as opposed to small particles prevailed by volume and characterize by low number contents. It should be noted that large particles have disappeared after dissolution of dried arnica extract. Volume and numerical distributions of 1 µm and 3 µm particles have been changed. After dissolution the volume content of small particles have increased in several times. Numerical content have decreased for 3 µm particles in 5 times. In arnica extract solution the presence of 150 nm particles by DLS have been detected. These particles are prevailed by number content.
Mallow dried extract was very polydispersive. Particles with diameter 1-2 µm present only one size group by number. Volume distribution was presented by several groups.
In sample of hyporamin solution a presence of 3 size groups have been found. 50 µm and 150 µm particles prevail by number. 150 µm and 200 µm particles have the same high volume contents. Hyporamin solution has get monodispersive due to an occlusion in plaster mass. Only 200 µm particles after dissolution of hyporamin plaster mass have been detected. It is a basic precondition for uniform distribution of dried extract particles. The uniform distribution of particles depends on biological activity of drugs and very important in technology of application dosage forms. This particular is very important for transdermal therapeutic patches such as plasters and for viscous pharmaceutical preparation e.g. ointments and gels.

4. Conclusions

For the first time the laser diffraction technique was applied for analysis of herbal extracts. In this paper we offer a new approach in standardization and quality control of herbal extracts. Particle size distribution is most important characteristic for particulate systems. Identification of particle size distribution by LALLS and DLS techniques is well grounded due to total statistical estimation of samples. All three types of plant extracts have been analyzed show different size spectra of volume and numerical distribution. This peculiarity can be used in establishing of plant extracts quality. Combination of LALLS and DLS with scanning electron microscopy demonstrates good complementarity of mentioned techniques. It should be noted that laser diffraction is pharmacopeial method (USP, EPh) and gradually introduces into practice.

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