

MEASURING THE MICROSTRUCTURE OF NATURAL GUMS

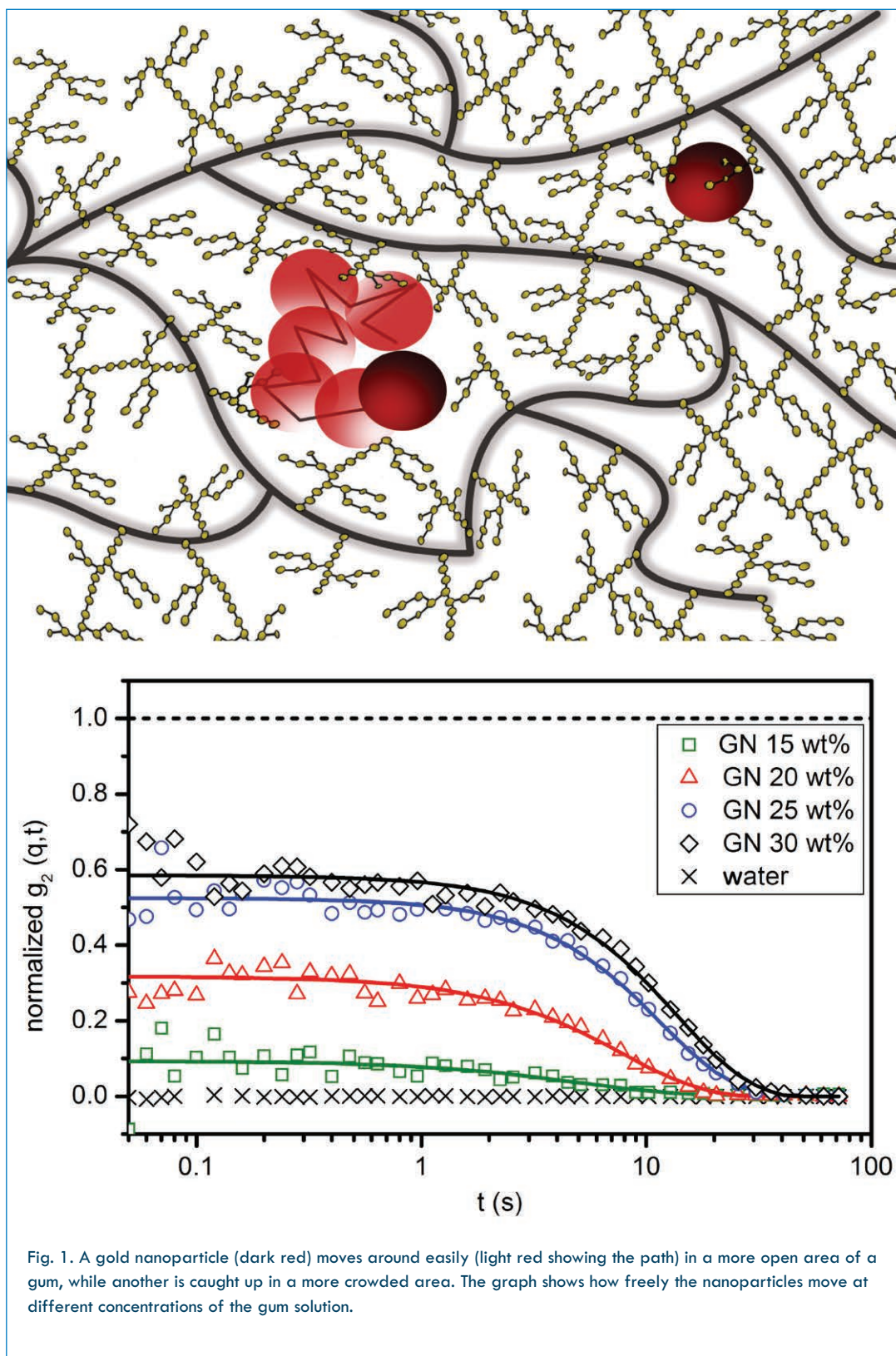


Fig. 1. A gold nanoparticle (dark red) moves around easily (light red showing the path) in a more open area of a gum, while another is caught up in a more crowded area. The graph shows how freely the nanoparticles move at different concentrations of the gum solution.

Accacia gum is widely used in foods, pharmaceuticals, cosmetics, and inks as a stabilizer or thickening agent. Usually the gum, a natural polymer, is derived from two main species of acacia trees, *A. senegal* and *A. seyal*, but there are roughly 1200 species of acacia. People who harvest the trees commercially would like to know how the gums from different species, which have different chemical variations, compare, so they can predict their material properties and their usefulness in different applications. Now scientists working at the APS have shown they can use x-rays to characterize the structural properties of the gum and relate those to how the material behaves. Measurements of these and other gums may help commercial users of the material decide which type is best for a given application. Meanwhile, these researchers say their results demonstrate that x-ray photon correlation spectroscopy (XPCS) is a viable method for studying the structure and dynamics of natural polymers.

The researchers from Argonne, Universidade Federal do Paraná (Brazil), Instituto de Pesquisa Pelé Pequeno Príncipe (Brazil), and Johns Hopkins University studied gum derived from *A. mearnsii*, harvested from trees grown for tannin extraction in Brazil. The gum from that tree was known to have better emulsifying properties and different viscosities from the commercially available gum. The gum has three main components: the biopolymer arabinogalactan, an arabinogalactan-protein complex, and glycoprotein. Understanding the arrangement of these components and the spacing among them gives a hint to the macroscale rheological properties of the gum—how it stretches, recoils, or flows as forces are applied in a certain direction.

X-rays provide the nanometer-scale resolution necessary to measure the structure, but the gum, consisting mainly of carbon and nitrogen, does not scatter x-rays well, so investigators could not see the structure directly. To overcome this problem, they synthesized gold nanoparticles and mixed those into the polymer. By seeing how the nanoparticles moved, they could get an idea of how the average spacing in the gum structure changed as the gum concentration was varied.

The researchers first measured the macroscopic behavior of the gum, and used that to predict how much space would be available at the nanoscale to let the nanoparticles move freely. They then made x-ray measurements of both the commercial and the Brazilian gum, which turned out to agree with their predictions. Increasing the ratio of gum to

water in the solution thickens the gum and reduces nanoparticle movement, a result that is more evident in the Brazilian gum, suggesting the components are becoming more entangled and leaving less space (Fig. 1). That matches the behavior at the macroscale, in which the Brazilian gum become more viscous more quickly. Overall, the shear viscosity—a measure of how resistant a substance is to flow—is about 1000 times lower in the commercial gums than in the Brazilian gum.

To understand how the nanoparticles fit with the gum's molecules, researchers needed to know their size distribution. They performed small-angle x-ray scattering on the nanoparticle solution at the XSD 12-ID-C,D beamline to determine that the average nanoparticle diameter was 4.8 ± 0.8 nm. That beamline has a large-area detector that can record the scattering from a wide range of nanoparticle sizes. The measurements were consistent with TEM images taken at the Argonne Center for Nanoscale Materials, which showed the gold nanoparticle's average diameter was 5.0 ± 1.2 nm.

To characterize the gums, they performed XPCS at the XSD 8-ID-I beamline. That beamline uses a coherent x-ray beam sensitive to the arrangement of the particles and how that varies over time, allowing researchers to watch how the particles move. The instrument can look at changing speckled interference patterns from the material that are sensitive to motion at the nanoscale, providing the resolution necessary for the experiment. — *Neil Savage*

See: Aline Grein-Iankovski^{1,2}, Izabel C. Riegel-Vidotti², Fernanda F. Simas-Tosin^{2,3}, Suresh Narayanan¹, Robert L. Leheny⁴, and Alec R. Sandy^{1*}, “Exploring the relationship between nanoscale dynamics and macroscopic rheology in natural polymer gums,” *Soft Matter* **12**, 9321 (2016).

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Author affiliations: ¹Argonne National Laboratory, ²Universidade Federal do Paraná, ³Instituto de Pesquisa Pelé Pequeno Príncipe, ⁴Johns Hopkins University

Correspondence: * asandy@anl.gov

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8-ID-I • XSD • Polymer science, materials science, physics • X-ray photon correlation spectroscopy, intensity fluctuation spectroscopy, small-angle x-ray scattering • 6-12.5 keV, 7.35-7.35 keV, 7.35 keV • On-site • Accepting general users •

12-ID-C,D • XSD • Chemistry, physics, materials science • Small-angle x-ray scattering, grazing incidence small-angle scattering, wide-angle x-ray scattering, surface diffraction • 4.5-40 keV • On-site • Accepting general