After 90 years, a better way to measure the composition of paper

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Researchers at the National Institute of Standards and Technology (NIST), in collaboration with the U.S. Government Publishing Office (GPO), have developed a novel, nondestructive method to rapidly measure the wood and non-wood fiber components in paper.

Identifying and measuring the ratio of plant fibers used to manufacture paper has wide application in criminal forensics, conserving art, authenticating historical documents, assessing the content of recycled paper and ensuring that passports and other U.S. government documents are printed on the requisite security paper.

For example, high-quality government documents are often created with non-wood fibers such as cotton. Wood-derived fibers make paper more brittle over time and can help reveal its age. Forensic investigators at a crime scene often look for the transfer of materials between individuals; such materials include the kinds of fibers that are in paper.

Despite its importance, the current method of analyzing paper has changed little since fiber technologist Mary Rollins of NIST (then known as the National Bureau of Standards) helped pioneer the method in the 1920s and 30s. By modern standards, however, the technique is laborious, time-consuming and highly subjective. The process also requires sacrificing a portion of the paper sample, which may be limited and needed for evidence.

To liberate individual fibers in the sample, the paper must be boiled in water, macerated (softened) with a glass stirring rod and treated with several chemicals. Then an eyedropper full of the fiber solution is placed on a microscope slide to dry. Next, iodine stains the fiber to render it visible. Then, the analyst must rely on his or her memory and visual acuity to match the shape of the stained fibers to textbook images of some 100 plant fibers.

NIST scientists Yaw Obeng, Jan Obrzut and Dianne Poster, along with NIST guest researcher Michael Postek and their colleague Mary Kombolias of the GPO, have now brought the fiber analysis of paper into the 21st century, using a method recently used to examine the material aging in microelectronics devices on semiconductor chips.

Their measurements can be performed in minutes and leave the entire sheet of paper intact.

The technique, known as dielectric spectroscopy, identifies the composition of materials by examining how particular molecules respond to a rapidly changing electric field. In adapting the technique to paper, the researchers focused on the behavior of water molecules, which are added during the manufacturing process and are also a key component of the plant fibers used to make paper. (Water molecules are a small but important constituent of dry paper.)

Microwaves shining on a sheet of paper induce the molecules to rotate. The rate at which water molecules rotate in paper differs from the rate they would rotate in free space. That's because the water molecules in the fibers are bound to naturally occurring polymers and other materials in the paper, which affect the rotation rate. The specific frequency at which the water molecules rotate therefore provides a clue about the chemical environment of the water molecules and therefore the content of the paper.

Water molecules provide excellent probes of the composition of the paper in which they reside. Water is a polar molecule, meaning that its positive and negative charges are slightly separated from each other. As a result of this separation, one end of a water molecule has a positive charge while the other end has a negative. When an alternating electric field is applied to the paper, the polarity of the water molecules aligns with the direction of the electric field. When the field reverses direction, which happens many billions of times a second, the water molecules try to follow suit, reversing their polarity in sync with the field. But the match isn't perfect.

That's in large part because the response of the water molecules depends on the composition of the paper—specifically the nature of the polymers to which the water molecules are bound. For instance, lignin, a polymer in plant cell walls that makes plants rigid and woody, will significantly slow the rate at which the water molecules can flip their orientation when an alternating electric field is applied. Recording the response rate of the water molecules therefore provides a highly sensitive measure of the type of plant fibers and their concentration in a paper sample.

"How quickly the water molecules adjust to the alternating electric field tells us a lot about the composition of the paper," Obeng said.

The researchers reported their findings in a recent issue of *Tappi Journal*, which includes research on forest products and related industries.

The team, along with other scientists, is now exploring how the same method might be used to detect harmful bacteria on surfaces in hospital rooms, and on recently caught fish and other perishable foods. The technique may work because just like water molecules, some bacteria have a distinctive way of reorienting themselves in the presence of an alternating electric current and relaxing when the current is switched off.

More information: Broadband Dielectric Spectroscopic Studies of Biological Material Evolution and Application to Paper. www.nist.gov/publications/broa … terial-evolution-and

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