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Exploiting Analytical Instruments to Address Water Crises

Toward Optimal Strategies for Sound Water Cycle

In a nation with natural water resources as abundant as Japan's, the threat of water shortages may seem distant—and yet, without a vigilant protective mindset, the risks that existing water sources may deteriorate in quality, or even run dry, are all too real. Meanwhile, the progress of global warming has created a host of increasingly dire problems facing water-supply systems around the world. It is against this backdrop that Professor Hiroshi Yamamura, of Chuo University's Department of Integrated Science and Engineering for Sustainable Societies of Faculty of Science and Engineering, harnesses advanced analytical instruments to pursue the elusive goal of *sound water cycle*.

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A three-pillared vision for sound water circle

The *Sustainable Development Goals* (SDGs), adopted by the United Nations General Assembly in 2015, are a set of international benchmarks designed to spur progress around the world toward solutions for thorny global problems, including environmental degradation, bias and discrimination, poverty, and threats to human rights. There are a total of 17 SDGs, and one of them is: *Clean Water and Sanitation: Ensure availability and sustainable management of water and sanitation for all*. As we will see in this article, the central theme of Yamamura's research—*sound water circle*—seems almost custom-designed to address this challenge.

At present, Yamamura's laboratory is pursuing a research program based on a three-pillared strategic vision.

The first pillar is **robust water circulation**: recognizing that water is a blessing from nature and the source of all life, taking care not to extract more water from nature than is strictly necessary, and purifying contaminated water before returning it to nature.

Rural societies in today's Japan are facing an existential threat: "People living near rivers, lakes, and other bodies of water tend to develop cultures and lifestyles that are closely tied to the water," Yamamura explains. "However, as Japan continues evolving into an elderly society with fewer and fewer young people, the pool of cultural curators capable of carrying on those traditions is rapidly dwindling, leaving regional water resources unmanaged—and creating the possibility that water-circulation networks could simply disappear. The key question is how to bequeath our water resources—and the regional cultures surrounding them—to future generations."

The Japanese countryside is dotted with towns famous for their subterranean water bodies and natural springs. In recent years, however, some of these have begun to run dry due to overuse and other factors; without a keen awareness of the preciousness of these resources, and a commitment to preserving them, the risk that underground water sources could suddenly degrade in quality—or vanish altogether—is very real. Moreover, Japan's accelerating evolution into a society of senior citizens with few children—and the corresponding depopulation of the countryside—creates a need for closed water-circulation systems contained entirely within regional boundaries. "This is why we're trying to design geographic regions capable of using and reusing water indefinitely," Yamamura explains, "and why we need to redesign regional societies to incorporate vigorous cultural traditions of water-resource management.

The second pillar is **harnessing water infrastructure to protect human life and ensure lifestyle convenience**. In Tokyo and other metropolitan areas that cannot rely on natural resources such as underground water bodies, safe and secure management of water infrastructure is essential. For citizens to trust the drinking water that comes out of their taps, water-quality information must be easily available for anyone to access.

"Japanese water-treatment plants use chemicals to purify water by inducing precipitation of impurities," Yamamura explains. "But the volume of chemicals used differs from day to day. Right now, the way it works is that inspectors at the plant look at the state of the water each day and say, 'Hmmm, it looks a little cloudy today—let's add a little more of the chemicals than usual.' However, if we could replace this with some sort of AI-based monitoring system going forward, that would mitigate problems caused by the shortage of maintenance personnel, and might even allow us to set up automated systems for supplying clean water in developing countries. So we're researching techniques for making tap-water quality more visible, and for implementing smart automated control systems for water-supply networks."

The third and final pillar of Yamamura's research vision is **establishing solid-liquid separation technologies**. *Solid-liquid separation* is just what it sounds like—the challenge of separating solids from liquids—and is a key requirement for any successful water-circulation system, which must remove various forms of solid matter, including numerous organic substances present in waste water and sludge, from the water they circulate. Yamamura's efforts in this area include efforts to optimize operating procedures for solid-liquid separation membranes.

A first imperative: understanding the behavior and properties of organic substances with various molecular weights

Let's dive more deeply into the nature of Yamamura's research.

Rivers, lakes, and other natural water bodies contain many varieties of organic substances; a first key step in achieving robust water circulation is to identify the types and origins of the organic compounds present in a given water source.

"Broadly speaking," Yamamura explains, "the organic substances present in natural water bodies can be divided into two categories: those derived from animals, and those derived from plants. In both cases, the substances have enormous molecular weights when they are first released into the water, but they gradually decompose over time due to sunlight exposure, microorganisms, and other factors. This decomposition process yields highly unsaturated forms of organic matter known as *humic substances* (Figure 1), which exhibit a broad range of molecular weights and comprise many different varieties of constituent components. In fact, the ingredients comprising humic substances vary dramatically from one water body to another—

and from one season to the next. Highly unsaturated humic substances tend to bind with metals and form complexes, which play a major role in cycling matter through the natural world—for example, by facilitating the transport of iron. However, during the purification process used to prepare drinking water, chlorine—added as a disinfectant—is known to react chemically with humic substances to produce the carcinogen trihalomethane. For this reason, understanding the behavior and properties of humic substances—both in nature and in industry—is of paramount importance."

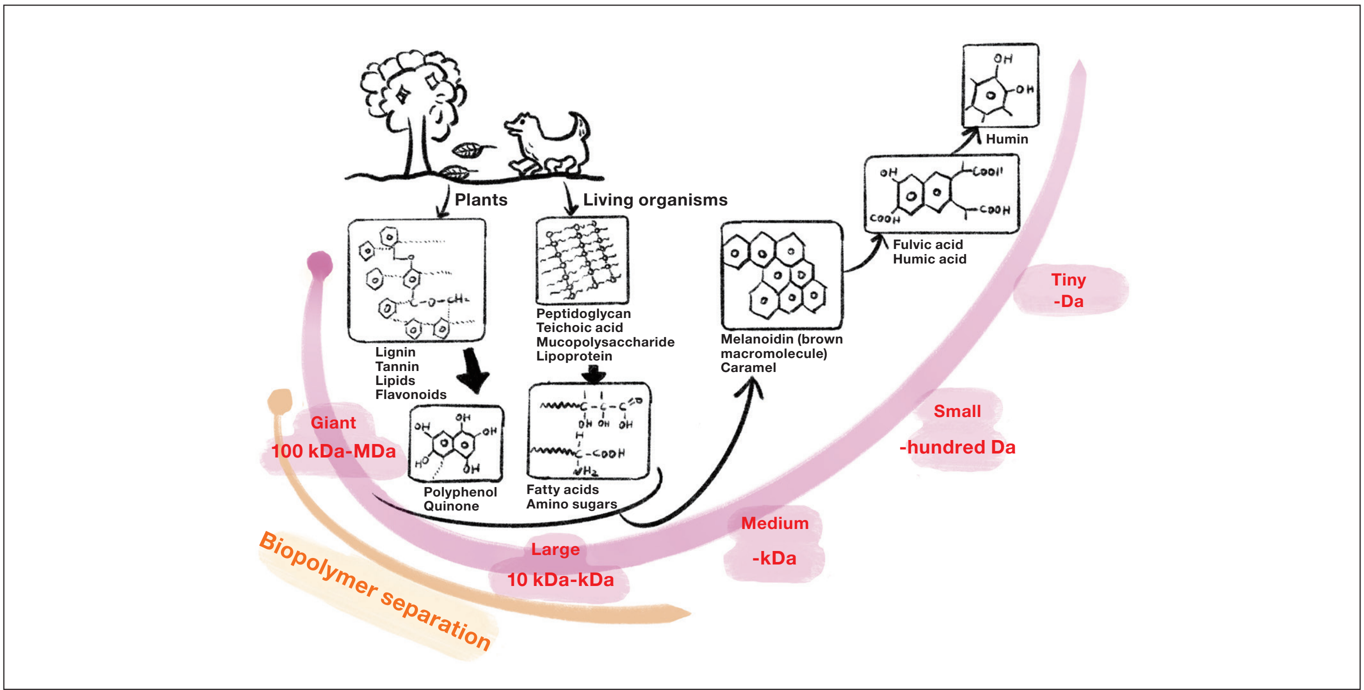

Fig. 1 Process of organic substances in natural bodies of water.
Organic substances in natural bodies of water may be divided into two categories: animal-derived and plant-derived. In both cases, the substances initially have enormous molecular weights, but gradually decompose under the influence of sunlight, microorganisms, and other factors, yielding highly unsaturated forms of organic matter known as *humic substances*. Humic substances exhibit a broad range of molecular weights and are comprised of many different varieties of constituent components.

Fig. 2 Researchers in Yamamura's laboratory use analytical instruments to identify substances contained in water samples collected both in Japan and overseas. These photographs were taken during a sample-gathering expedition in Japan.

Researchers in Yamamura's laboratory collect water samples from various geographical regions both in Japan and overseas (Figure 2), then analyze these samples in different ways: using ion-exchange resins to separate and refine humic substances, then using analytical instruments to identify fulvic acid and other constituents contained in these humic substances.

"In the past," says Yamamura, "the primary tools used to analyze these substances would be mass analyzers, but this would mostly involve high-precision analysis of specific substances. Mass analyzers designed to study low-molecular-weight organic compounds are, needless to say, ideal for analyzing small molecules, but are not well-suited for studying things like humic substances, which comprise many different types of ingredients. For analyzing natural water bodies containing humic substances, it seemed clear to me that we really needed a new type of instrument capable of measuring many different types of organic substances—spanning a broad range of molecular weights—systematically and with high sensitivity."

These considerations led Yamamura to focus on a different type of instrument: *fluorescence spectrophotometers*, which use measurements known as *fluorescent fingerprints* to enable rapid and easy analysis of humic substances and other samples considered difficult to study via conventional techniques.

Fluorescent fingerprints are obtained by measuring fluorescence intensity over a range of excitation and fluorescence wavelengths, then plotting intensity vs. wavelength in the form of a contour map; this yields a data visualization that tends to resemble a human fingerprint—whereupon the name.

Inside a fluorescence-emitting sample, electrons typically exist in minimal-energy orbitals known as *ground states*. When light shines on the sample, electrons absorb energy and transition to higher-energy orbitals known as *excited states*; when these electrons later return from the excited state to the ground state, they emit energy in the form of light, which is observed as fluorescence from the sample.

In contrast to conventional fluorescence measurements, in which the excitation light is fixed at a single wavelength, fluorescent-fingerprint measurements vary both the excitation and fluorescence wavelengths over given ranges while measuring the fluorescence intensity

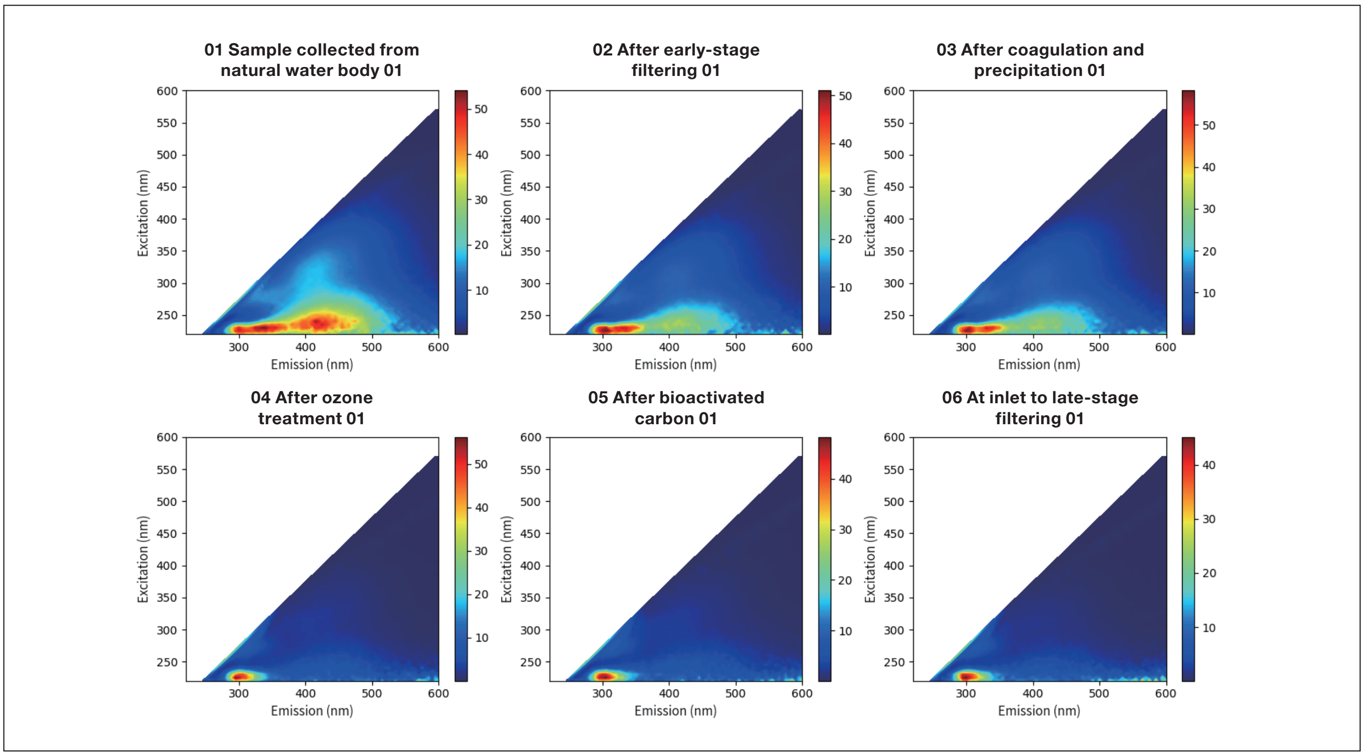

Fig. 3 Examples of three-dimensional fluorescent fingerprint spectra.
These fluorescent fingerprints were measured for a sample of water sampled from a natural water body and subjected to various purification steps, including ozone treatment. In the fluorescent fingerprint for the unprocessed sample, we see significant fluorescence intensity over a wide range of fluorescence wavelengths between 300 and 600 nm, indicating the presence of a diverse variety of organic substances with widely-varying molecular weights. In contrast, no similar intensity features are seen for ozone-treated sample, indicating that the treatment process successfully removes most organic substances.

from a substance. The resulting fluorescent fingerprint—a three-dimensional contour plot of measured intensity vs. excitation and fluorescence wavelengths—is a unique visual pattern that is characteristic of the substance. This allows systematic determination of the various fluorescent substances contained in a sample—even for complex samples such as foodstuffs in which multiple constituent components are blended together (Figure 3). Fluorescence-emitting substances are abundant in the natural world. For example, many organic compounds present in foodstuffs—such as chlorophyll and tryptophan—are fluorescent, and fluorescence spectrophotometers were thus conventionally used to measure concentrations of chlorophyll and similar substances. This led Yamamura to wonder if similar techniques could be used to analyze humic substances contained in water environments.

Fluorescence spectrophotometers: Enjoying a well-deserved recent turn in the spotlight

"Actually, the fact that fluorescent substances have unique, characteristic fluorescent fingerprints was understood already by around 1990," Yamamura recalls. "But, although people did recognize the minuscule variations in fluorescent-fingerprint data, there was really no way to exploit them—the field had to wait another 20 years before it became possible to extract all the information derivable by analyzing measured data. However, in recent years we've seen improvements both in data-analysis methods and in the performance of desktop computers, and now we can use a technique called *multivariate analysis* to determine the substances present in a given sample quickly and easily simply from fluorescent-fingerprint measurements. Multivariate analysis is a strategy for statistically processing datasets encompassing large numbers of variables. Also, in the past few years we've seen some new data-analysis techniques based on artificial intelligence (AI), which are pushing the

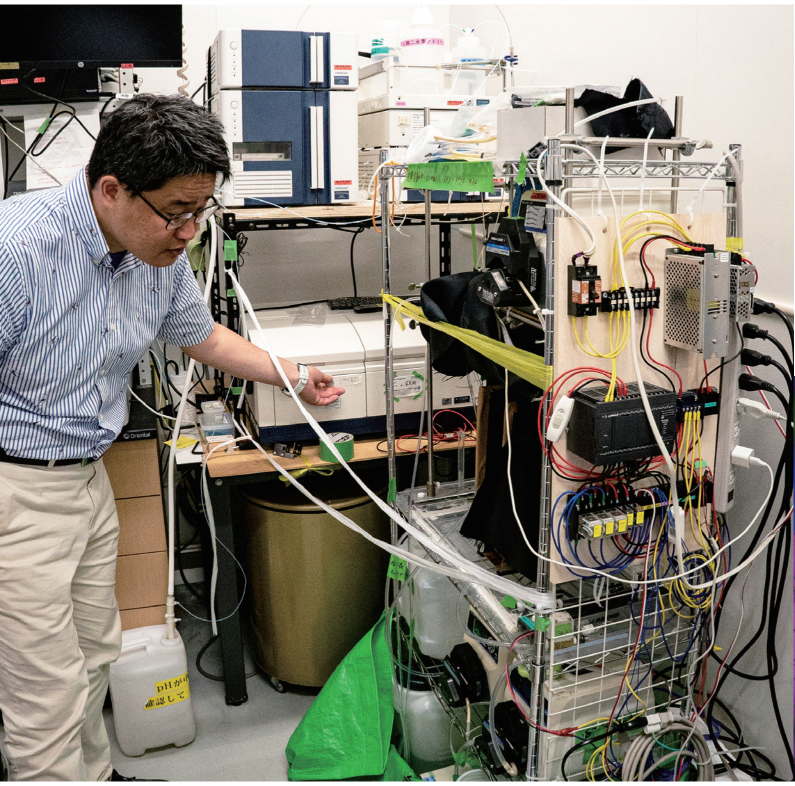

Fig. 4 Yamamura's monitoring system for separation membranes.
The system uses a specialized optical fiber, designed by Yamamura, to monitor the state of separation membranes. These membranes tend to clog after repeated use, degrading performance; Yamamura's system monitors this behavior to select times for automated membrane cleaning.

Fig. 5 A culture tank for cultivating microalgae.
This enormous tank, which occupies an entire corner of Yamamura's laboratory, is used to cultivate microalgae. These particular microalgae species are capable of producing oil, making them a focus of recent interest for biofuel applications.

field into the spotlight once again."

These motivations led Yamamura to add a new instrument to his laboratory: an F-7100 fluorescence spectrophotometer from Hitachi High-Tech. "When I was a student," Yamamura remembers, "a single fluorescent-fingerprint measurement took as long as an entire hour. In contrast, our F-7100 can acquire one fingerprint *per minute*. That's an incredibly dramatic improvement."

Fluorescent-fingerprint measurements proceed by irradiating the sample with light spanning a broad range of frequencies, from the optical to the ultraviolet, then detecting the resulting fluorescence from the sample to yield a three-dimensional dataset describing *fluorescence intensity* vs. *excitation wavelength* and *fluorescence wavelength*. This dataset typically consists of several thousand measured data points. However, generating high-resolution fluorescent-fingerprint images requires irradiating the sample with light at a larger number of wavelengths, which can be time-consuming; the F-7100 has the dual advantages of performing rapid measurements and offering high-resolution mapping functionality.

"In one of our research papers," Yamamura recalls, "we wrote 'Our instrument acquires data for 1000 wavelengths per second,' and the referee reviewing our paper thought this must be a typo—'Did you mistakenly write an extra zero in that number?' he asked. This tells you something about the F-7100—it can make measurements at speeds that would have been unthinkable just a short while ago."

The result was that a task that was once prohibitively difficult—identifying the types and origins of the constituent components of humic substances—became possible. "At the end of the day," Yamamura muses, "this is what's so fascinating about analytical techniques—they let you see things that were previously invisible. The F-7100 has had a revolutionary impact on my research."

Using fluorescence spectrophotometers to study separation membranes

Yamamura has recently begun tackling a new challenge: using fluorescence spectrophotometers to study separation membranes.

"There's one problem with separation membranes," Yamamura admits, "that has bothered me ever since I was student." His laboratory collects water samples from rivers and lakes in Japan and other countries, then uses separation membranes to remove solid matter—containing humic substances—from these samples. However, the process of sampling and analyzing organic substances trapped in separation membranes requires tearing the membranes apart, after which they cannot be reused. "So, one day I was talking with a friend at Hitachi High-Tech, and I mentioned how convenient it would be if we could use fluorescence spectrophotometers to measure organic substances in separation membranes *directly*, without needing to disassemble the membranes. And that was when my friend brilliantly suggested using optical fibers!"

This proposal was inspired by an earlier project in which Hitachi High-Tech researchers had used optical fibers for fluorescence analysis of pigments and dyes in the process of restoring cultural artifacts. If an optical fiber connected to an F-7100 could be used to make direct measurements of organic substances in separation membranes, there would be no need to disassemble the membranes. "And, when we actually tried it out," Yamamura recalls, "we were amazed—sure enough, just as my friend had predicted, making measurements with optical fibers allowed us to acquire highly accurate data. My many years of frustration were over!"

Yamamura exploited this technique to develop a system for monitoring the state of separation membranes using optical fibers. These membranes become clogged with repeated use, degrading their performance and requiring periodic cleaning procedures involving specialized chemicals; however, carrying out this cleaning step *too* frequently can shorten membrane lifetimes and increase operating costs. Yamamura's monitoring system automatically determines optimal cleaning schedules—and automates the actual cleaning process as well (Figure 4).

Thus Yamamura's approach to analytical instruments is not simply to purchase, install, and forget about them, but rather to devote considerable time and effort to making improvements and adding new functionality as necessary. "Whenever I talk to the folks at Hitachi High-Tech," Yamamura notes, "they always offer tons of helpful suggestions—and they even work together with us to add new features to existing systems. I consider Hitachi High-Tech to be an essential partner in my research."

The ongoing evolution of analytical instruments —and their future trajectory

"These days," notes Yamamura, "analytical instruments are evolving into more than just boxes in which you mount samples and make measurements." Following the example of his separation-membrane monitoring system, going forward Yamamura expects that each and every analytical system will eventually be augmented by control systems that can automate operation based on analytical results. "For example, when it comes to monitoring the quality of drinking water at water-treatment plants, analytical instruments have traditionally been little more than quality-management tools," Yamamura explains. "However, going forward we'll be able to connect to operational control systems at prior stages of the water cycle, which will allow more advanced quality controls. I'm also excited to see applications of AI."

A similar evolution is underway at chemical plants and food-processing facilities, which are in the process of becoming *smart factories*. "In the future," Yamamura predicts, "people will be using analytical instruments for more and more purposes, and I think the questions of how to make effective use of the data they generate—and how to use these systems to give something back to society—will be major themes."

A pair of water shortages at a formative age —and a lifelong source of inspiration

What was the original impetus for Yamamura to start researching water-circulation systems? "I grew up in Japan's Kagawa Prefecture," he begins, "and, in Japan, Kagawa is synonymous with *udon* noodles. Water is essential for making *udon* noodles, but it turns out that Kagawa has relatively few natural springs—instead, we had always purchased water from our neighbors in Tokushima Prefecture. Anyway, when I was in middle school, there were two occasions on which we experienced water shortages, and in each case we were told that a truck would be coming around to deliver water to each household. So we raced down to the store to buy plastic water jugs, but when we got there they were almost totally sold out—and what they had left had been marked up to exorbitant prices! These experiences left me with a visceral understanding of two things: first, that people can't live without water, and second, that water can actually be a big business! I'd say that was the origin of my interest in water-circulation research."

Later, as a student at Hokkaido University, Yamamura began research on filter membranes capable of producing clean drinking water by filtering sea water or waste water. "I was thinking that the ability to transform sea water into drinking water would instantly solve all of Kagawa's water problems—but it was right around that time that they build a dam, which solved all the problems before I ever got the chance. I've always felt a little upstaged by that dam!" Yamamura chuckles. "Nonetheless, I found research on membranes to be totally fascinating, and that's how I got to where I am today."

Yamamura finds one key advantage of water-circulation research to be the breadth of its overlap with other fields of science and engineering, which has led him to collaborate with researchers in areas he never imagined could be relevant to his work. One recent example is a collaborative project to study certain species of oil-producing microalgae, which have attracted interest for possible biofuel applications; Yamamura's laboratory is studying separation membranes for separating the microalgae from their culture liquid. This has led to the installation of an enormous culture tank, occupying an entire corner of the laboratory, in which the microalgae are cultivated (Figure 5).

Yamamura's goals for the future are to establish sustainable water-circulation protocols to facilitate carbon-neutral societies: "I'd love to propose something like a self-contained water-circulation system that can exist entirely inside one single-family home."

Yamamura is also committed to solving water problems in developing countries. "For example, right now I'm involved in a project using 3D printers to make filter membranes for water treatment in Tanzania, Africa," he explains. "If these filters could be made on-site using 3D printers, there would be no need to import them all the way from Japan, and each region could make filters appropriate for their unique needs. The hope is to make it easy to convert muddy river water into clean drinking water."

In the battle to combat global warming and tackle the dire problems facing water systems around the world, Professor Hiroshi Yamamura's fighting days have only just begun.

(Reported and written by Kumi Yamada. Photographs by Takashi Horigome.)