Cellulose optical fiber for sensing applications

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ABSTRACT

Cellulose materials offer new biodegradable alternatives for fabricating optical fibers for sensing applications. These environmentally friendly materials have intrinsic properties that existing glass and polymer fibers lack. Cellulose fibers are hydroscopic and thus can rapidly take liquids in and also dry quickly. Cellulose based optical fibers can be manufactured from regenerated cellulose or cellulose derivative that offers a large property space. They can be resistant or dissolves in water, and refractivity index of the material can be tuned as needed. In this work, feasibility for sensor applications of three different cellulose optical fibers have been tested: regenerated cellulose for water and humidity sensing, carboxymethyl cellulose (CMC) for respiratory rate monitoring, and methylcellulose (MC) for short-range 150 Mbit/s signal transmission at 1310 nm. This fast signal transmission can be utilized with short cellulose-based sensor fibers. The work shows the scientific potential of a novel optical material for photonics.

Keywords: Cellulose, optical fiber, regenerated cellulose, carboxymethyl cellulose, methylcellulose, respiratory rate

1. INTRODUCTION

Optical fibers, a linear structure that can guide light, have been available a long time. The first glass optical fiber (GOF) was demonstrated with Kao and Hockham 1966 [1]. Later the lowest attenuation value 0.2 dB/km was shown and GOF came the main signal transmission technology for optical telecommunication [2]. Next the durable and easy to use polymer optical fibers were invented [3]. These fibers have been used to short distance signal transmission with few hundred meters inside buildings. Installation of POF fibers is little bit easier, but higher attenuation 12 dB/km [3] limits applications to short distance applications.

GOFs have been used also for sensor applications for long time. Optical time domain reflectometry (OTDR) measures Rayleigh backscattering to monitor attenuation of GOF as function of the distance [4]. Fiber optic distributed temperature sensing (DTS) is powerful technique to measure temperature as a function of distance with standard GOF [5]. DTS is based on Raman scattering in the optical fiber. DTS can measure temperatures with 1 m distance resolution using a few kilometers long fiber. Fiber Bragg grating (FBG) makes possible to measure temperature and strain [6]. It is possible to do several FBGs to the same fiber and create sensor network even several kilometers long fiber. There is fiber interrogator equipments that can measure several optical fibers at the same time. OTDR, DTS and FBG are all used to structural monitoring applications like bridges [7] and oil/gas drilling [8]. Separate sensor device could be also connected to the end of the GOF. For example, the pressure has been measured optically with silicon membranes that have been integrated to the end of optical fibers [9]. Pressure has been monitored also with the external membrane in a journal bearing with reflection type fiber optics sensor [10].

POFs have been used for heath sensor applications. Leal-Junior et al. measured breath and heart rate simultaneously [11]. They increased the sensitivity by removing cladding and a part of the fiber core. Sartiano and Sales used POFs to monitor movement and respiration with cutted fibers type sensor [12]. It is also possible make FBG to POF. Liu et al. demonstrated thermally tuned FBG in PMMA POF [13]. Tuning range was more than 18 nm with 50°C heating, which is much more than tuning range of typical FOG FBGs.

GOF and POF are not biodegradable and it also difficult to modify the waveguide material itself. However liquid and gas sampling is possible with photonic crystal (PC) fibers. Both GOF [14] and POF [15] have PC fibers that have holes inside the fiber. These holes are used to create light guiding structure inside the fiber without higher refractive index core material. The same holes have been used to liquid [15] and gas measurements [16].

In this work novel cellulose based optical fibers were tested. These cellulose-based fibers are biodegradable and biocompatible. They are sensitive to water, because they are hydroscopic, and can take water in and also dry fast. These fibers do not compete with GOF and POF in telecommunication, but they can offer new measurement possibilities to sensor applications. Some applications were demonstrated to verify the benefits and possibilities of the cellulose optical fibers. Regenerated cellulose (RC) fiber [17] was used to water and humidity measurements with a loop type sensor. Respiratory rate was measured with carboxymethyl cellulose (CMC) fiber [18] with a reflection type fiber probe. Short distance signal transmission was demonstrated with methyl cellulose (MC) fiber [19].

2. METHODS

2.1 Manufacture of optical fibers

RC optical fiber was prepared by using the dry-jet wet spinning [17]. In general, the cellulose dissolved in EMIM[OAc] was passed through a nozzle to water bath that regenerated the fiber followed by drying in air. CMC optical fiber were prepared by using dry-jet wet spinning [18]. In general, CMC dissolved in water was spun with a nozzle to water bath containing aluminum sulphate followed drying in air. Heat treatment 10 min. in 160°C was used for CMC fiber to make it water resistant [18]. Heat treatment makes cellulose yellower [20] and it also improve infrared transmission [18]. MC optical fiber was prepared by using wet spinning [19]. In general, MC dissolved in cold water was spun into ethanol and dried in air.

2.2 Transmission measurements

Light transmission was measured with RC fiber in a water bath and in a humidity cabin (SH-221. Espec). Single mode (SM) fiber (9/125 µm core/cladding diameter) was coupled from 1310 nm SLED (S5FC1021P, Thorlabs) to RC fiber. Transmitted light was measured with multimode (MM) fiber (200/220 µm core/cladding diameter) and indium gallium arsenide (InGaAs) photodetector (S155C, Thorlabs) with a power meter interface (PM101, Thorlabs). Light coupling to RC fiber was done with commercial FC/PC connectors, adapters and ferrules (Figure 1). LabView software was used to collect signal from photodetector. In water measurement just 1-3 cm long part in the middle of the RC fiber was in water. In humidity measurement full length of the RC fiber was inside the humidity cabin.

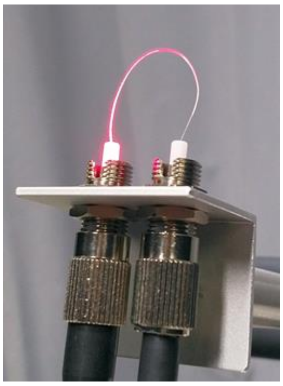

Figure 1. RC fiber with commercial FC/PC connectors.

2.3 Transmission spectrum measurements

Transmission spectrum was measured for MC fiber. Halogen lamp (HK-2000-HP, Ocean Optics) was coupled with MM (105/125 µm core/cladding diameter) to MC fiber. Transmitted light from MC fiber was collected with MM fiber (400/425 µm core/cladding diameter) to an Optical Spectrum Analyzer (OSA) (AQ-6315A, Ando). Light coupling to MC fiber was done with micromanipulators (Melles Griot) to avoid direct light coupling between light source and detector [17, 18]. OSA measured transmission spectrum from 350 to 1750 nm with 10 nm resolution.

2.4 Spectroscopic measurement for red food color

Spectroscopic measurement was done with 637 nm laser (S4FC637, Thorlabs) and 1050 nm super luminescent light emitting diode (SLED) (S5FC1050P, Thorlabs) in Figure 2a. Wavelength was selected with 2x2 switch (Dicon). SM fibers (9/125 µm core/cladding diameter) were used to couple light from light sources to RC fiber and MM fiber (400/425 µm core/cladding diameter) collected transmitted light to silicon (Si) photodetector (S120C, Thorlabs) with a power meter interface (PM101, Thorlabs). LabView software was used to collect signal from photodetector. Red food color was diluted to water in concentrations 0.1, 1 and 10%. RC fiber was 45 mm long and just 1 cm long part in the middle of the RC fiber (Figure 2b) was in the sample solution.

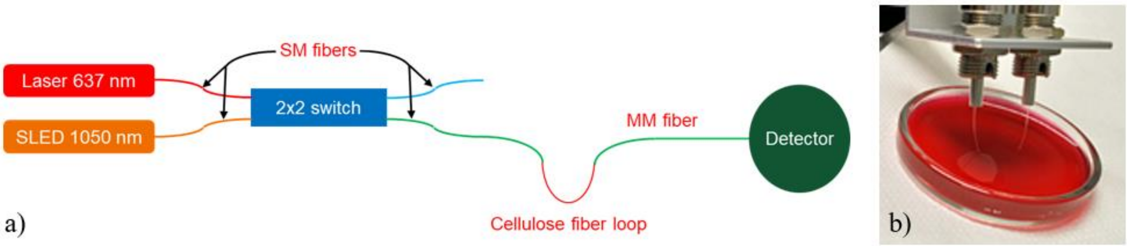

Figure 2. a) Spectroscopic measurement setup and b) RC fiber loop in the sample solution.

2.5 Reflection measurements

Respiratory rate was shown with reflection type optical fiber probe (Figure 3a). SM fiber (9/125 µm core/cladding diameter) and MM fiber (105/125 µm core/cladding diameter) were glued and polished to ferrule with 270 µm inner diameter. Sleeve and another ferrule with 340 µm inner diameter were used to couple CMC fiber to reflection probe (Figure 3b). SLED (S5FC1050P, Thorlabs) with 1050 nm wavelength was used as light source and Si photodetector (S120C, Thorlabs) with a power meter interface (PM101, Thorlabs) for light reflected light measurement.

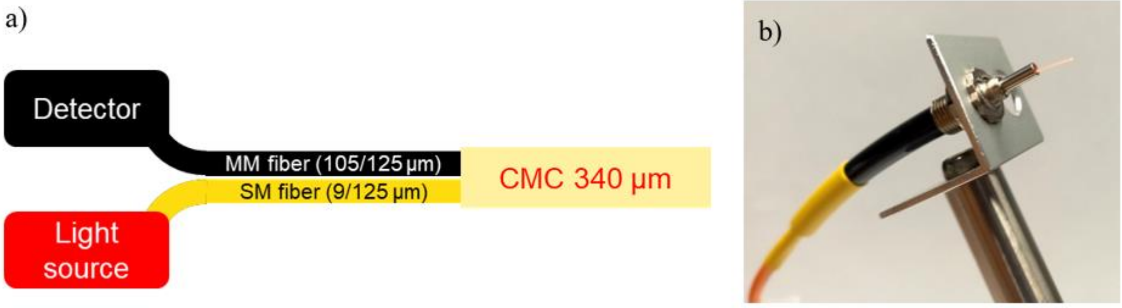

Figure 3. a) Reflection measurement setup and b) light coupling to CMC fiber.

2.6 Short distance signal transmission

Short-range broadband signal transmission through the cellulose fiber was demonstrated using MC fiber. The schematic of the setup is shown in Figure 4. Light from a distributed feedback laser (TSL-510, Santec) at 1310 nm wavelength was modulated using an external modulator (OC-192, JDSU). The modulation pattern a 150 Mbit/s non-return to zero (NRZ) pseudo random bit sequence (N = 2^15-1) data stream was created using a pulse pattern generator (D3186, Advantest D3186). The light was coupled to the MC fiber using a graded index MM optical fiber (62.5µm/125 µm core/cladding diameter) with 0.275 numerical aperture (NA) to ensure that the light wakes several guiding modes in the MC fiber. The light output from the MC fiber sample was collected via a step index MM glass fiber (400/425 µm core/cladding diameter) with 0.39 NA to a fixed gain amplified large-area photodetector (InGaAs, PDA05CF2, Thorlabs). Output of the photodetector was connected to a sampling oscilloscope (CSA8000, Tektronix) that was triggered by the signal from the pulse pattern generator. The obtained eye pattern was used to evaluate the functionality of the short-range MC fiber link with multimode connection fibers.

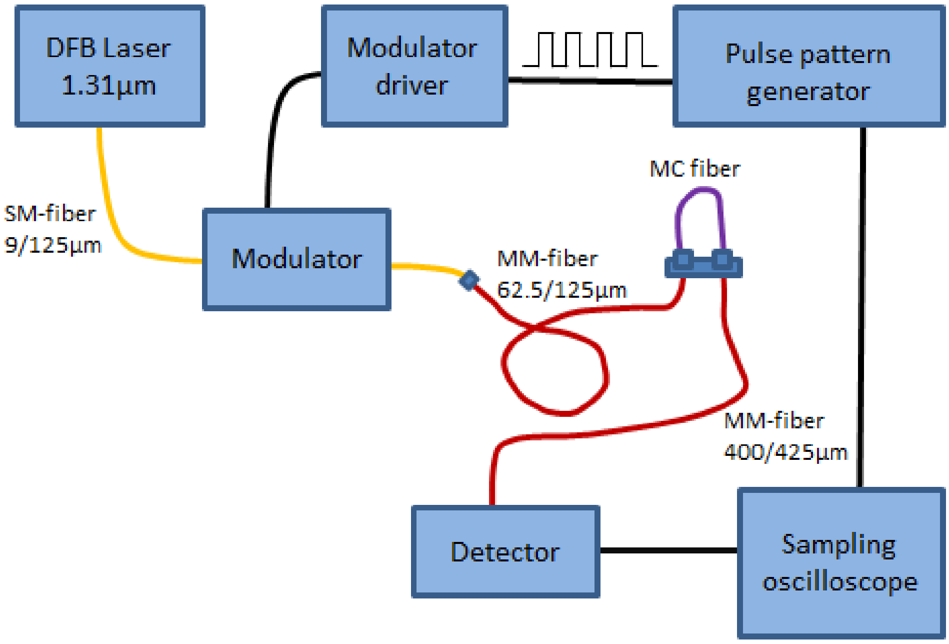

Figure 4. High-speed optical signal transmission measurement setup for the MC fibers.

3. RESULTS

3.1 Water measurements with regenerated cellulose fiber

RC fiber wetting was measured in water bath with transmission setup. Different fiber lengths in middle of RC fiber loop were in water (Figure 5). Transmission decreased 10, 19 and 22 dB as a function of the wetted fiber length 1, 2 and 3 cm, respectively. Total length for the fibers were 41, 52 and 60 mm for 1, 2 and 3 cm wetting, respectively. All samples were first 10 min. in water and then 10 min. in air. Wetting and drying both took about 4 min. The transmission change is not instant, because cellulose water took water inside the fiber. Light transmission decreased due to high attenuation of water at 1310 nm wavelength range.

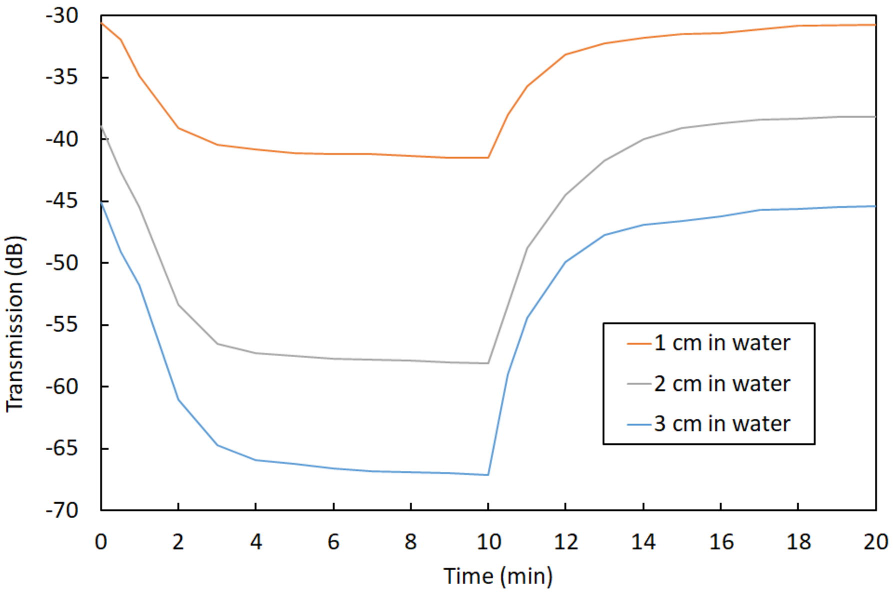

Figure 5. RC fiber wetting in water.

3.2 Humidity cabin measurements with regenerated cellulose fiber

RC fiber wetting was measured also in the humidity cabin with transmission setup using the same 52 mm long RC fiber as above in the water bath. The signal change with the same fiber in water bath was 19 dB with 2 cm wetting length. Now the signal change was just 2.3 dB with full 52 mm length when RH change from 50 to 90% (Figure 6). The time constant was also much longer. The signal did not stabilize even in few tens of minutes. For example, in higher 90% RH signal did not stabilize in 45 min. and in lower 50% RH it took about 25 min. before signal stabilized. Humidity ramps at 60, 70 and 80°C were 15 min long, but results showed that temperature ramps should be much more than 45 min. That would mean very long measurement period, because now the total time for this experiment was 3 hours and 40 min. Temperature was constant 25°C during the whole measurement period.

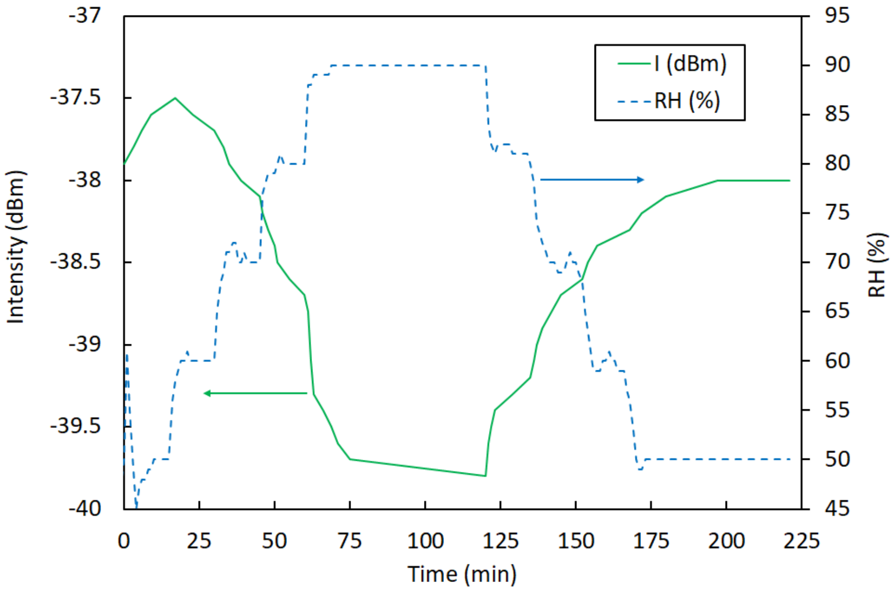

Figure 6. RC fiber length 52 mm wetting in the humidity cabin.

3.3 Red food color sampling with regenerated cellulose fiber

RC fiber was measured with spectroscopic measurement setup using red food color solutions 0-10%. Red food color has absorption just in 637 nm wavelength area, but water absorption happens in both wavelength areas 637 and 1050 nm. For that reason, 637 nm laser act as signal wavelength for red food color sampling and infrared 1050 nm SLED act as reference for water drying. Both wavelengths were measured at the same time with the same sample. In Figure 7a&b, the RC fiber loop was first 10 min. in red food color solution and then 17 min in air. In Figure 7a, absorption at 637 nm increased from 0.5 dB to 1.1, 1.6 and 2.5 dB with 0.1, 1 and 10% sample solutions, respectively. In Figure 7b, absorption at 1050 nm recovered to 0.5±0.25 level after 6 min. drying. Variation of the end level comes from incomplete drying or light coupling. Absorption increase at 637 nm as a function of red food color concentration shows that it is possible to use RC type cellulose fiber as a sampling device for water applications.

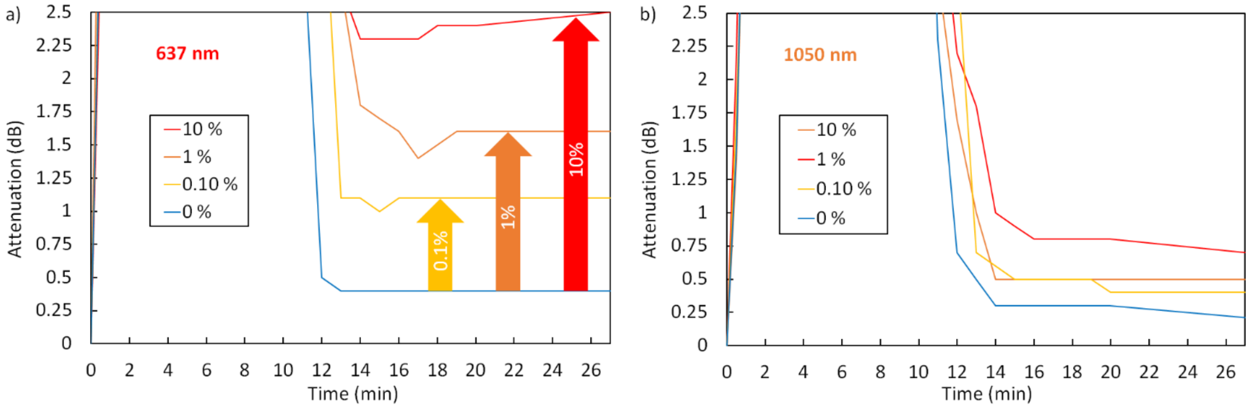

Figure 7. Red food color absorption with RC fiber at a) 637 nm and b) 1050 nm.

3.4 Respiratory rate measurement with CMC fiber

Reflection setup was used for respiratory rate measurement in Figure 8. Breathing was done towards the end of the 19 mm long CMC. Breathing signal has three phases in Figure 8. First the signal decreased due to water droplet in the end of the CMC fiber. Second water droplet dried and the signal increased higher than original ground level, because the end surface roughness of CMC fiber has been polished by water. Third CMC fiber end dried in just over a second, and the signal decreased to original ground level fast enough before a next breath. Measured respiratory rate in Figure 8 was 39 breaths per minute.

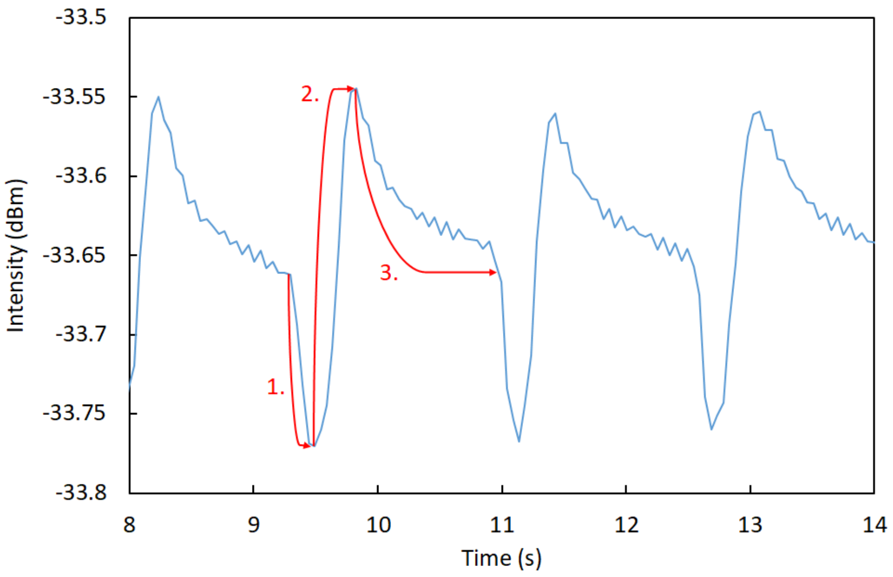

Figure 8. Respiratory rate measurement with CMC fiber reflection: 1. water droplet on the fiber end, 2. water inside the fiber and 3. fiber drying to ground level.

3.5 Short range signal transmission with MC fiber

The measured transmission spectrum of the MC fiber is shown in Figure 9. The fiber transmit light in the wavelength range from 600 nm to 1350 nm. The lowest attenuation is at about 1100 nm wavelength. Transmission peak is also seen at around 1300 nm wavelength. Similar type transmission spectra have been measured before for the other types of cellulose fibers [17, 18].

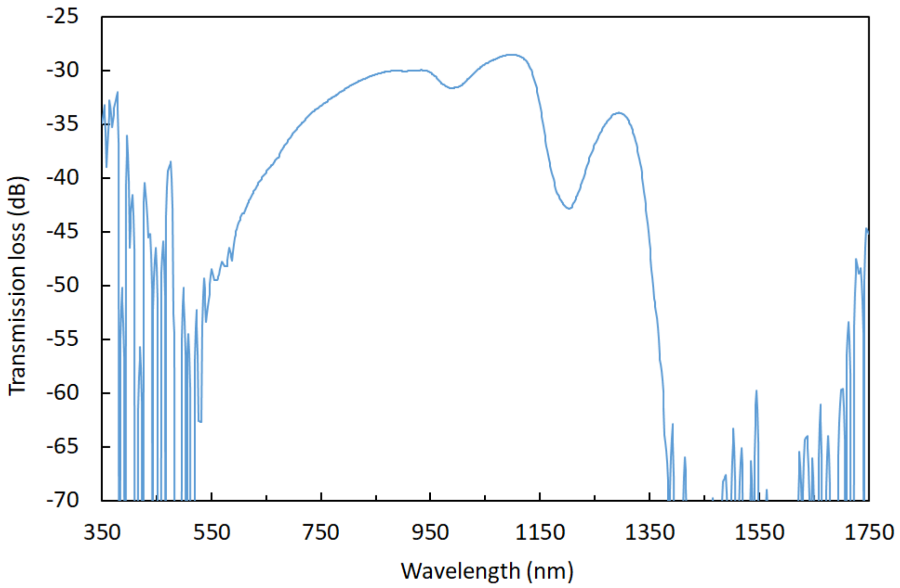

Figure 9. Transmission spectrum of MC fiber.

The maximum optical signal transmission capability of a 4.3 cm long MC fiber was tested using high-speed signal transmission setup. The wavelength was chosen to be 1310 nm because it is typical wavelength in telecommunication applications and measuring devices were available for that wavelength. Signal transmission experiments in the other 1550 nm telecommunication wavelength range are not possible because the transmission of MC fiber is between 550-1400 nm. The tested MC fiber was uncoated, and the core size was 300 µm. Figure 10 illustrates the measured eye patterns from the signal transmission measurements. Constant binary levels 1 and 0 are clearly distinguishable and the transitions between the levels are well seen from the measured eye patterns, which corresponds to minimal signal distortion in the optical link. Observed about 3 ns rise and fall time comes mainly from the detector.

Due to the large diameter, a wide-area detector must be used, whereby the bandwidth of the detector (150 MHz) limits the achievable transmission speed. Higher speed can be achieved by reducing the core size of the MC fiber or accepting a higher coupling loss with the detector. However, achieved 150 Mbit/s a high signal transmission rate is already sufficient for many sensing applications. If we compare speed to telecommunication applications for instance 150 Mbit/s rate is sufficient to run six Ultra HD 25 Mbit/s television channels in parallel.

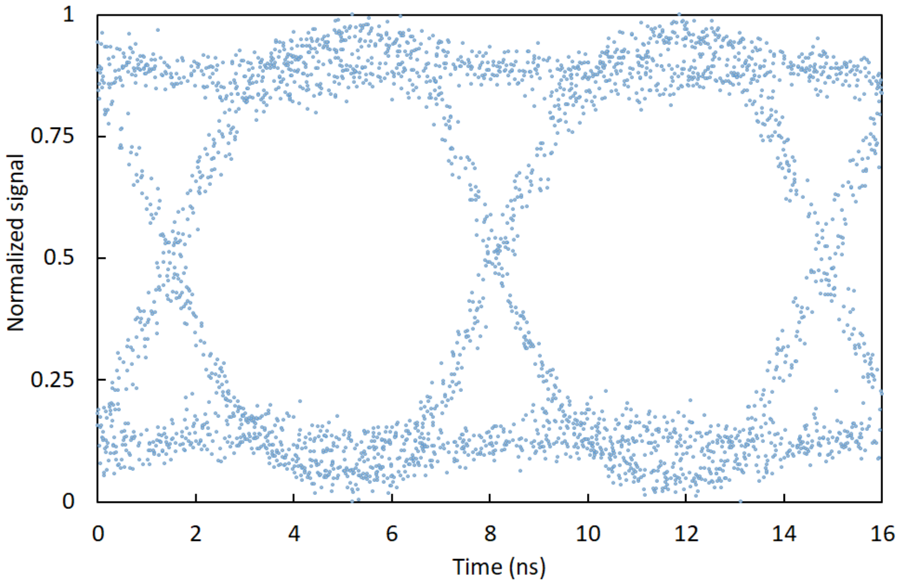

Figure 10. Recorded eye diagram of optical signal transmission through MC fiber.

4. CONCLUSIONS

Wetting properties of RC fiber was tested in the water bath, in the humidity cabin and with red food color sampling. RC fiber took fast liquid in and also dried fast in water bath experiments. Wetting and drying steps were 10 min. long, but already in 4 min. both wetting and drying were almost completely saturated. Water attenuation at 1310 nm was 10-21 dB with 1-3 cm wetting length, respectively. RC fiber wetting in humidity cabin was correspondingly very slow and signal did not saturate even in tens of minutes. Maximum attenuation increase was 2.3 dB with 5.2 cm long RC fiber that was completely inside humidity cabin. Red food color sampling was demonstrated with RC fiber. Attenuation after drying at 637 nm increased from 0.6 to 2 dB with 0.1-10% sample solution, respectively. Drying of the RC fiber was measured at 1050 nm, because there water has high absorption and dried red food color inside RC fiber does not increase the absorption. Respiratory rate 39 breaths per minute was shown with CMC fiber. Breathing was done towards the end of CMC fiber. Breathing increased air humidity and this change was measured. Drying was fast enough that signal recovers before next breath. Short distance signal transmission was demonstrated with MC fiber to show the optical performance of cellulose fibers. Maximum measured signal transmission rate 150 Mbit/s makes high-speed cellulose optical fiber sensors possible.

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