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Nonlinear optical modulation and ultrafast dynamics of Fe-BTC in a 2 μm thulium-holmium fiber laser

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Abstract

While Fe-BTC, a semiamorphous metal–organic framework (MOF), has been established for gas storage and catalysis, its application as a saturable absorber (SA) remains unexplored. In this work, Fe-BTC was successfully implemented as an optical modulator to generate ultrafast pulses in a thulium-holmium-doped fiber laser (THDFL). Fe-BTC was drop-casted on an arc-shaped fiber to form the optical modulator, yielding a modulation depth of 2.1%. With a 3-dB spectral bandwidth of 2.7 nm, the output wavelength was centered at 1896.7 nm. The repetition rate was 14.7 MHz, and the pulse width was 1.5 ps, corresponding to an optical peak power of 91 W and a pulse energy of 136 pJ. The system operated steadily, as evidenced by its signal-to-noise ratio (SNR) of around 55 dB. This study reveals the capabilities of Fe-BTC as a low-cost saturable absorber for ultrafast fiber lasers.

Introduction

Ultrafast fiber lasers have revolutionized numerous fields, from advanced manufacturing [1, 2] and telecommunications [3] to biomedical imaging [4, 5] and fundamental scientific research [6, 7], owing to their compact size, excellent beam quality, and robust operation [8]. The generation of ultrashort optical pulses in these systems is fundamentally enabled by mode-locking, which matches the phases of multiple longitudinal modes within the laser cavity. Among the various methods for achieving mode-locking, those that utilize a saturable absorber (SA) as an optical modulator are particularly attractive due to their passive, self-starting nature and simple design [9, 10]. A conventional SA exhibits intensity-dependent absorption, meaning that it becomes more transparent at higher optical intensities. This property favors the formation and amplification of short pulses while filtering out noise.

The characteristics of an SA significantly influence the properties of passively mode-locked fiber lasers. It includes modulation depth, saturation intensity, and recovery time. Conventional SAs, such as semiconductor saturable absorber mirrors (SESAMs), offer reliable performance but often suffer from complex fabrication and a narrow operating bandwidth [11]. This led to many studies on novel materials that can overcome these limitations, including low-dimensional materials such as graphene [12–14], carbon nanotubes (CNTs) [12, 13], and transition metal dichalcogenides (TMDs) [14–16]. While these materials have demonstrated promising saturable absorption properties, each has shortcomings, such as agglomeration during synthesis [17] and a low optical damage threshold [9].

This work introduces a new class of saturable-absorption materials: metal–organic frameworks (MOFs). MOFs are porous materials composed of inorganic metal clusters linked by organic linkers, yielding structures with exceptionally high surface area, tunable porosity, and diverse functionality [17–19]. Their unique characteristics, including bandgap engineering [20–22] and a high degree of control over their optical and electronic properties [23–25], make them ideal candidates for optoelectronic applications. A few studies have reported strong nonlinear optical absorption in MOFs [26–28] and have utilized it for ultrashort-pulse generation [29, 30].

Fe-BTC occupies a unique niche among MOFs due to its semi-amorphous (or semi-crystalline) nature, distinguishing it from the predominantly crystalline materials in this class. For years, it was widely used in the literature because it is commercially available (e.g., Basolite F300 by Aldrich/Merck). The direct (and sustainable) synthesis protocol for the semi-amorphous Fe-BTC [31] was first reported in 2015. This development improved the physicochemical properties of Fe-BTC, enabling the optimization of synthesis parameters. Structurally, it is closely related to MIL-100(Fe), one of the most studied MOFs due to its mesoporous structure, non-toxicity, low manufacturing cost, good redox properties, and sustainable preparation procedure [32]. Indeed, Fe-BTC and MIL-100(Fe) materials are equivalent at short- and medium-range scales, but the former lacks long-range order, whereas MIL-100(Fe) is crystalline. While Fe-BTC is well-studied in gas storage [26] and catalysis [27] applications, its usage as an SA at 2 μm is unexplored. Beyond its novel nonlinear optical potential, Fe-BTC's high thermal stability [28] makes it uniquely suited to withstand the significant thermal loads inherent in high-power 2 μm fiber laser systems. In short, when compared to other materials [33], its easy and sustainable synthesis process, commercial availability, and semiamorphous nature makes Fe-BTC a promising candidate for nonlinear optics applications.

Fe-BTC was synthesized by interlinking safe trivalent iron (III) and trimesic acid sources by following the method described in [31]. This Fe(III)-based MOF material exhibits catalytic activity comparable to that of the commercial equivalent, Basolite[®] F300 [34]. The prepared Fe-BTC is a MOF with a semi-crystalline structure and different physicochemical properties compared to MIL-100(Fe) [35].

Here, we report the successful integration of a MOF, Fe-BTC, into an SA for stable mode-locked pulse generation in a THDFL. We present the synthesis and characterization of the material and analyze the generated pulses. This work will open new avenues for exploring the vast diversity of MOFs in advanced photonics and ultrafast laser technology.

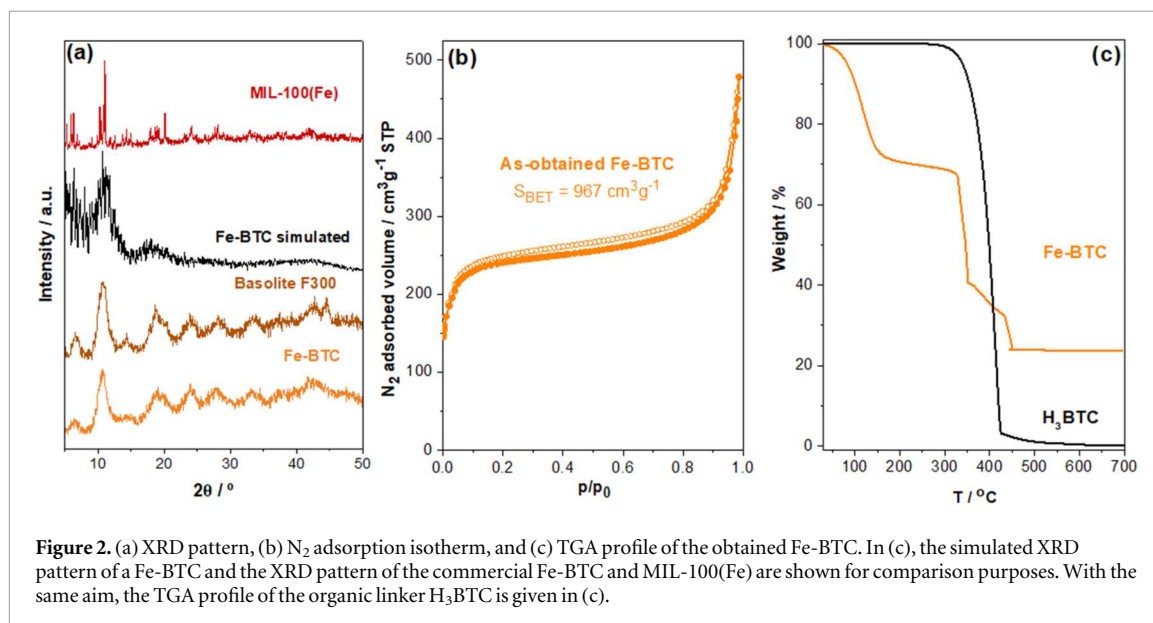
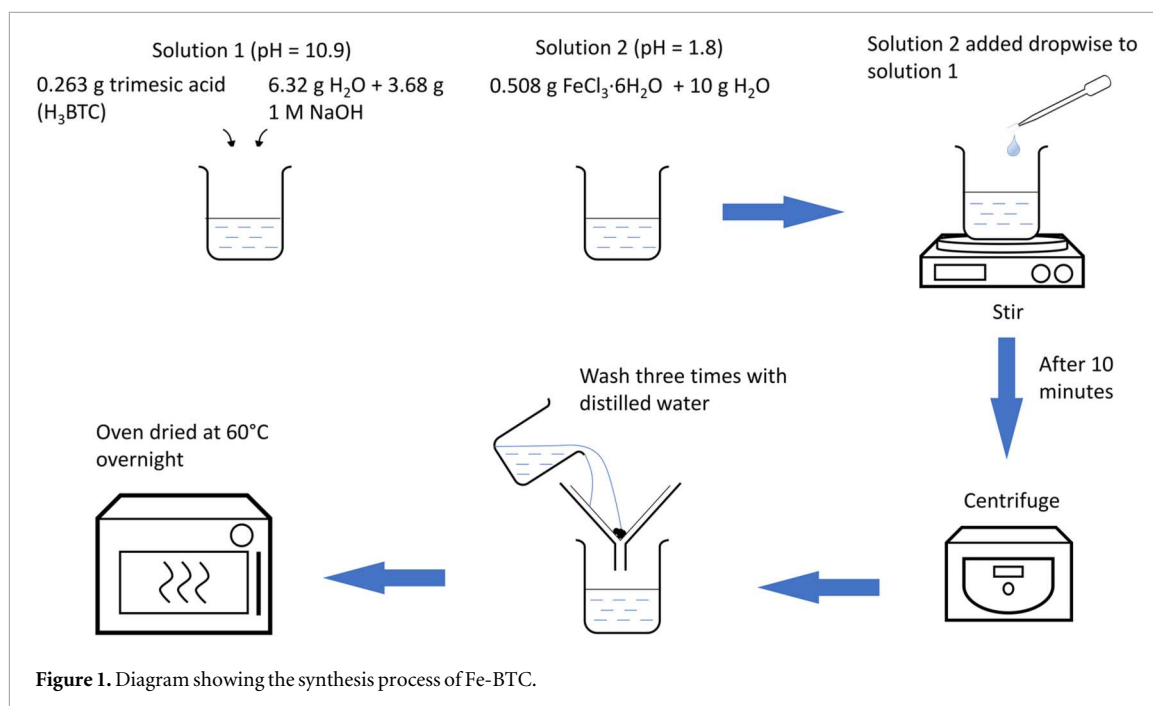
Synthesis and characterization of the FE-BTC

The Fe-BTC was synthesized as described in reference [35], and the process is illustrated in figure 1. Firstly, 0.263 g of trimesic acid (H_3BTC) was dissolved in 6.32 g of distilled water together with 3.68 g of 1 M NaOH aqueous solution (solution 1) as a deprotonating agent. Then, 0.508 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was dissolved in 10 g of distilled water (solution 2). Afterwards, solution 2 was slowly added to solution 1 under continuous stirring at room temperature, forming a brownish-orange precipitate almost immediately. The mixture was centrifuged for 10 min. Then, distilled water was used to wash it three times, followed by overnight oven drying at 60 $^\circ\text{C}$. The as-obtained MOF material was denoted as Fe-BTC. The fabrication and properties of Fe-BTC are reproducible, as it has been synthesized in previous works [27, 31, 32, 35–37].

Figure 2(a) compares the X-ray Diffraction (XRD) analysis pattern of the synthesized Fe-BTC with that of a commercial Fe-BTC Basolite F300, and the structurally-related MIL-100(Fe), as well as the XRD pattern simulated from the unique crystallographic information file (CIF) file available in the literature [34]. It must be noted that a non-crystalline material has no CIF file. That CIF file exists based on PDF (Pair Distribution Function) data [37], not on crystallographic data, which is used for crystalline materials. Indeed, despite a relatively good match between the experimental XRD pattern of Fe-BTC and the simulated one, the agreement is much better with the experimental pattern of the commercial Fe-BTC. A comparative analysis of the XRD patterns reveals that the Fe-BTC bands align slightly with the peaks of MIL-100(Fe), highlighting substantial structural similarities between the two.

Figure 2(b) shows the N_2 adsorption/desorption isotherms of Fe-BTC measured at -196°C . This material is observed to have a high specific surface area (965 m^2/g) and a pore volume (0.65 cm^3/g), both of which are higher than those reported in a previous study [35]. The semi-amorphous character of the Fe-BTC MOF material could eventually lead to non-negligible differences in textural properties.

The TGA (ThermoGravimetric Analysis) profile of the Fe-BTC sample is compared with that of the protonated linker H_3BTC in figure 2(c). The temperature at which the linker BTC^{3-} is lost from the FeBTC sample is significantly lower than that for the protonated linker, indicating that the linker changes its coordination in our sample relative to the organic linker source. The sample's thermal stability is consistent with previous research [31]. Density Functional Theory (DFT) calculations are an incredibly useful tool to analyze the properties of a



material, as shown in studies such as [38] and [39]. Although it was not performed in this study, it will be considered in future works.

Figures 3(a)–(d) indicates scanning electron microscope (SEM) images at different magnifications of the Fe-BTC sample. It is formed by shapeless micron-sized aggregates of nanosized spherical particles, which is in good agreement with a previously reported material synthesized under similar reaction conditions [31]. Since X-Ray photoelectron spectroscopy (XPS) is a technique that provides information about the surface of a material at a few nanometers, the data cannot fully represent the sample; they can only represent its surface. Additionally, XPS is not a routine technique for MOF characterization. Thus, XPS characterization was omitted.

Fabrication and characterization of the FE-BTC SA

The Fe-BTC SA was fabricated by drop-casting the SA onto the curved section of the arc-shaped fibre. The preparation of the arc-shaped fiber involves polishing a single-mode fiber (SMF) to remove part of its cladding, as described in a previous work [40]. It is done to allow the evanescent field outside the core to interact with the

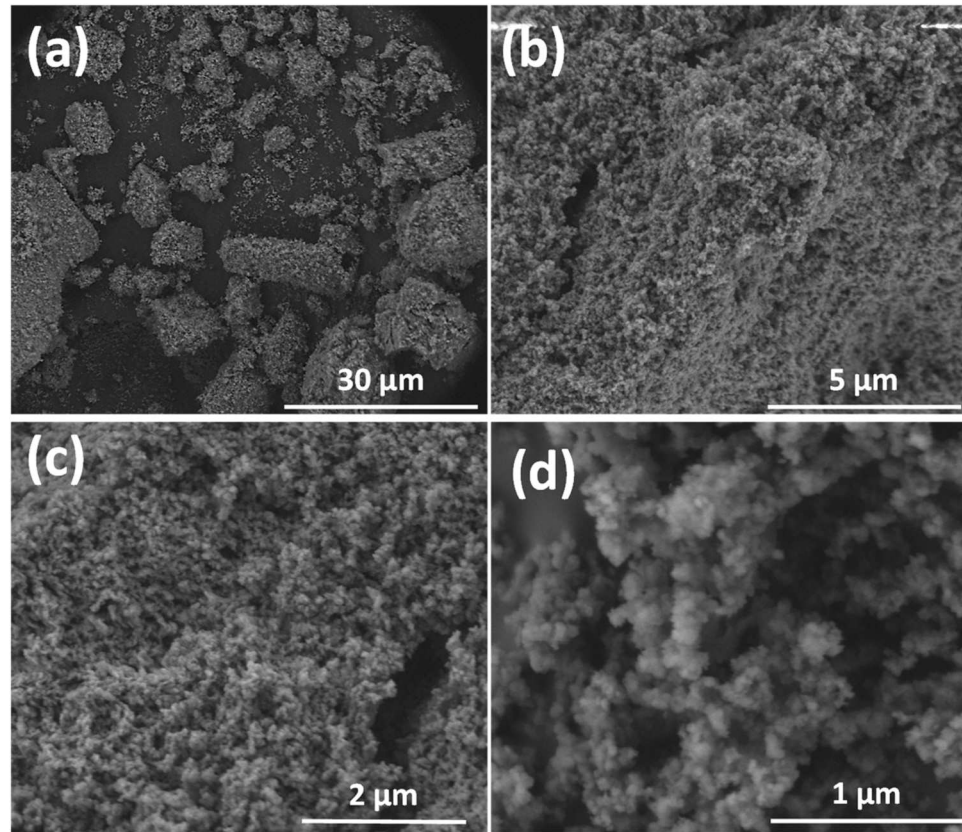


Figure 3. SEM images of the sample Fe-BTC at different magnifications: (a) 4000; (b) 24,000; (c) 50,000; and (d) 120,000.

Fe-BTC. After the arc-shaped fiber was drop-casted with Fe-BTC, it was allowed to dry at room temperature. Detailed dimensions of the arc-shaped fiber were given in figure 4.

Before the deposition of Fe-BTC onto the arc-shaped fiber, a linear loss of 70% was measured, as illustrated in figure 4(a). Following the drop-casting process, the loss increased to 78% due to absorption by the material, as depicted in figure 4(b). While most of the optical power is confined to the fiber core by total internal reflection, a small portion of the light travels in the cladding as evanescent waves. The fraction of power within the core, Γ , can be estimated with the equation,

$$\Gamma \approx 1 - \exp(-2a^2/w^2) \quad (1)$$

where a represents the core radius, and w represents the mode field radius [41]. According to equation (1), the estimated power in the core at 1896 nm is approximately 68%. As the cladding is polished away, the evanescent field is increasingly exposed to the surrounding medium. By coating the arc-shaped fiber with Fe-BTC, the evanescent wave was able to interact with the Fe-BTC, which is a requirement for pulse formation. If the material has a higher refractive index than the core, more light will be extracted from the core into the material. However, the lower refractive index of air ensures that some light is reflected into the core, maintaining guided propagation.

The SA exhibits a linear absorption of 6.6 dB at 1896 nm, as depicted in figure 5(a). The measurement was done using a THDFL amplified spontaneous emission (ASE) source. For measuring nonlinear absorption, a self-built mode-locked pulse laser source (PLS) was used, as shown in figure 5(b). The generated pulses had a pulse width of 1.02 ps, a repetition rate of 18.7 MHz, and a centre wavelength of 1955 nm. The output was adjusted with a variable optical attenuator (VOA) before being split by a 50:50 coupler, with one beam passing through the SA and the other serving as the reference beam. Both beams are measured separately using S148C optical power meters (OPMs) from Thorlabs, designated OPM 1 and OPM 2. Using the equation $T = 1 - \alpha_{ns} - \Delta T \cdot \exp(-I/I_{sat})$ [42], where T is the optical transmission, α_{ns} is the non-saturable loss, ΔT is the modulation depth, I is the peak intensity of the pulse, and I_{sat} is the saturation intensity. The measurement results are curve-fitted to the equation as shown in figure 5(c), giving a saturation intensity of 2.7 MW/cm^2 , modulation depth of 2.1%, and a non-saturable loss of 76.3%. Although the modulation depth is low, it is still sufficient for mode-locking operations as evident in previous works [43–45]. It is further supported by the numerical study by Jeon *et al* [46], which showed that the minimum modulation depth required

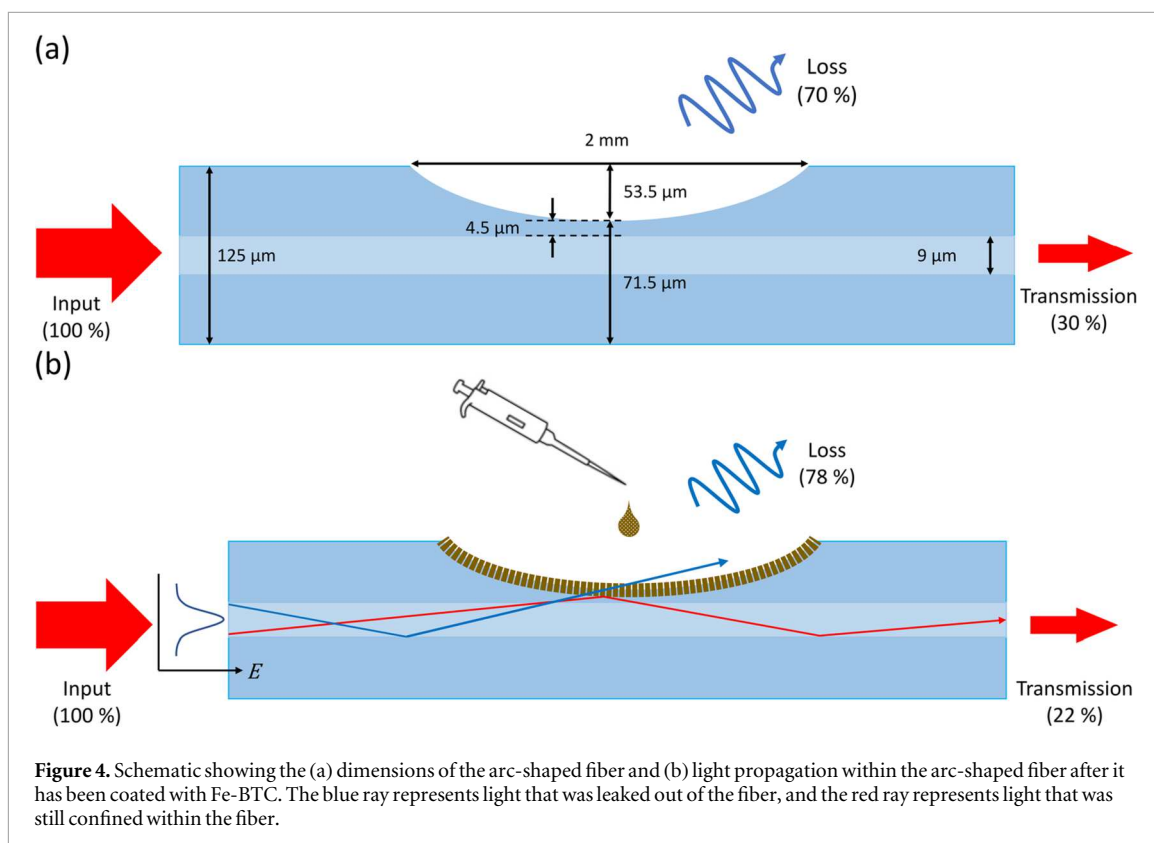


Figure 4. Schematic showing the (a) dimensions of the arc-shaped fiber and (b) light propagation within the arc-shaped fiber after it has been coated with Fe-BTC. The blue ray represents light that was leaked out of the fiber, and the red ray represents light that was still confined within the fiber.

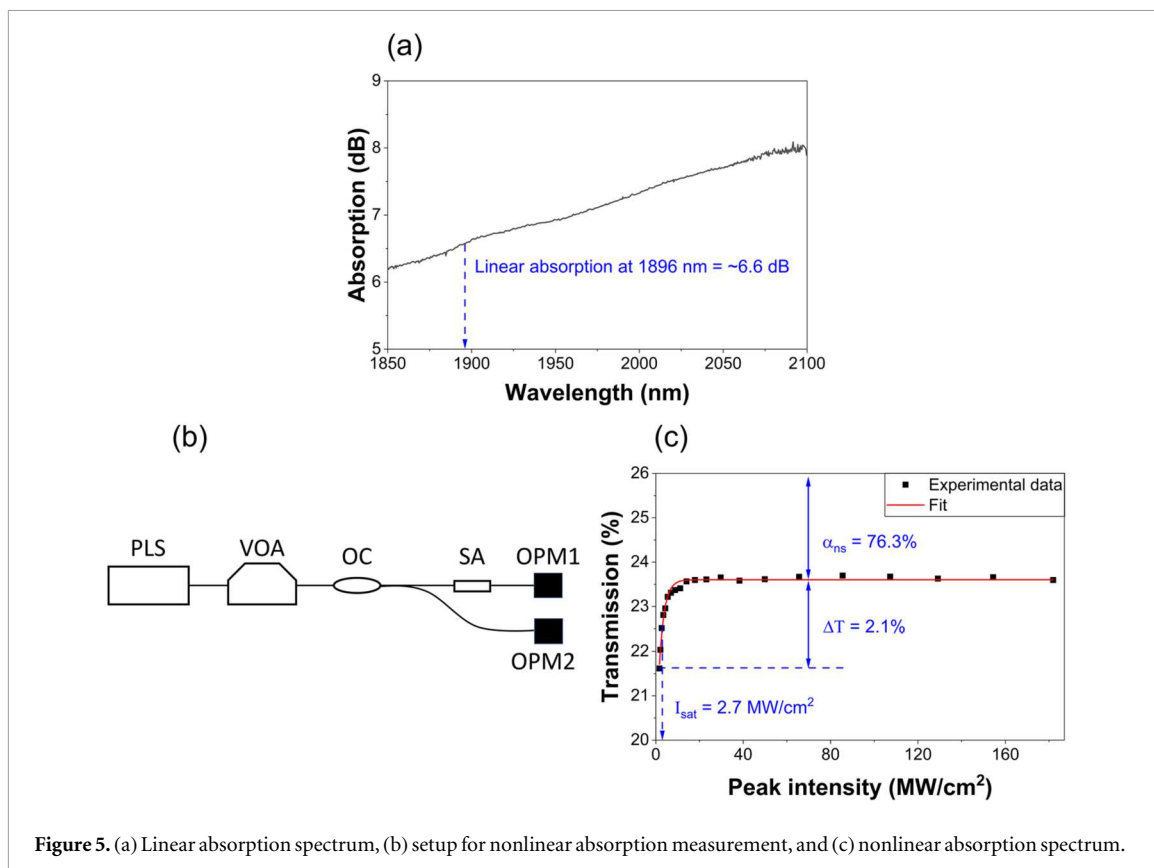


Figure 5. (a) Linear absorption spectrum, (b) setup for nonlinear absorption measurement, and (c) nonlinear absorption spectrum.

for mode-locking operation in a slightly anomalous dispersion regime is significantly smaller than that of normal or net-zero dispersion.

For an arc-shaped fiber, careful cladding removal enabled the evanescent field to interact strongly with the coated Fe-BTC, which is essential for mode-locked operation. However, this also introduces significant

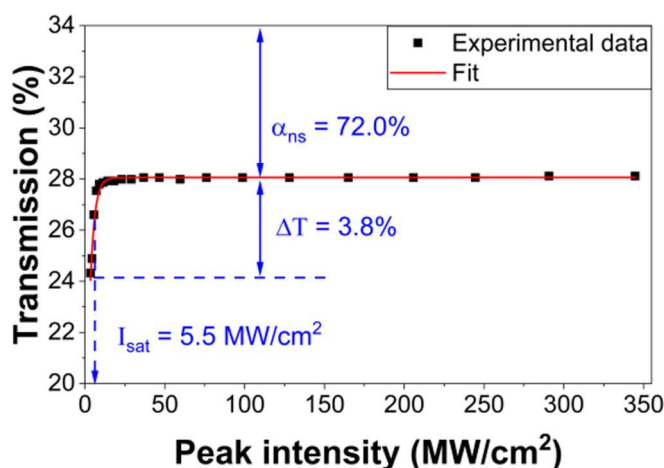


Figure 6. Nonlinear absorption spectrum of Fe-BTC SA at 1.5 μm .

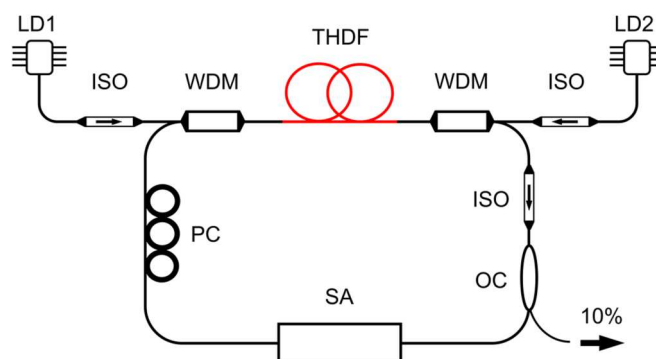


Figure 7. Schematic of the mode-locked THDFL using Fe-BTC SA.

scattering loss at the fiber-material interface. These scatterings, including Mie and Rayleigh scattering, may occur due to the physical imperfections of the system, such as rough interfaces at the fiber-material interface, bubbles in the fiber, or grain boundaries of the material. This loss is the primary contributor to the SA's non-saturable loss. Furthermore, because this SA is fiber-based, splice points may be another source of loss. It is because some of the light may leak into the cladding or be lost at splice points where the cavity connects to the SA. Another possible source of non-saturable loss is the linear absorption of non-active components at the operating wavelength. In the case of Fe-BTC SA, the main cause is the baseline absorption of the Fe-BTC.

The nonlinear optical response of the SA was also measured at 1.5 μm , and the results are shown in figure 6. The non-saturable loss of the SA is 72%, which is smaller than the loss at 2 μm (76.3%). It is expected because the insertion loss of the arc-shaped fiber at 1.5 μm is lower than at 2 μm , due to the smaller mode field diameter at 1.5 μm . The modulation depth of the SA at 1.5 μm is 3.8%, higher than at 2 μm , suggesting that the SA may operate at 1.5 μm . The higher saturation intensity at 1.5 μm of 5.5 MW/cm^2 compared to 2.7 MW/cm^2 may imply a higher mode-locking threshold at 1.5 μm . In short, this result reveals the potential of the Fe-BTC SA to function at 1.5 μm , and hints at the possibility of operating at other wavelengths. Further investigation will be conducted in future works.

Experimental setup

The THDFL used in this study was pumped by two 1560 nm laser diodes (LD1 and LD2) manufactured by Princeton Lightwave, as shown in figure 7. An optical isolator (ISO) was installed after each laser diode to minimize back-reflected light, greatly improving pump stability. Two wavelength-division multiplexers (WDMs), one for each pump, guide the pump lasers into the cavity while also confining the laser signals within it. The gain medium of the setup was a 1.5 m-long CorActive TH512 thulium-holmium-doped fiber (THDF) with a numerical aperture of 0.16 and a core diameter of 8.4 μm . The absorption coefficient is 15 dBm^{-1} at

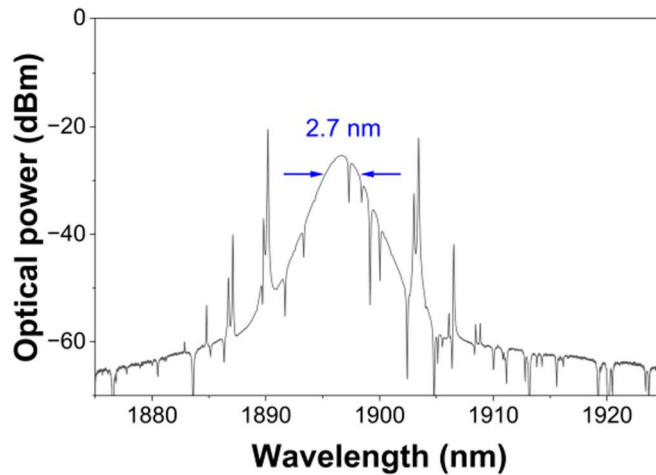


Figure 8. Optical spectrum of the mode-locked THDFL.

1550 nm. To ensure unidirectional operation, an optical isolator was needed. The 90/10 optical coupler splits the laser into two beams: the 10% beam is used for the output, and the 90% beam is fed back into the cavity. For the THDFL to operate in the mode-locking regime, fine adjustments to the polarization controller (PC) are required.

The active dopants in the gain medium are thulium and holmium ions. By co-doping, the gain medium can take advantage of thulium's absorption at 1560 nm and holmium's longer emission wavelength. Firstly, photons from the 1560 nm pump excite the thulium ions from the ground state, 3H_6 , to the excited state, 3F_4 . Once the thulium ions are excited, they do not necessarily emit light immediately. Instead, energy is transferred to holmium ions through a non-radiative process. Energy from the 3F_4 level of thulium ions is transferred to the 5I_7 level of holmium ions. It effectively populates the upper level of holmium. Then, the holmium ions at 5I_7 relax to the lower $5I_8$ level, emitting light at 1896.7 nm. Lastly, the emitted light underwent multiple round-trip through the cavity, amplifying the signal via stimulated emission, leading to lasing.

The cavity was 14 m long and comprised of the 1.5 m long THDF. As this is an all-fiber setup, the remaining 12.5 m of SMF comprises the fiber between components in the cavity and within the components themselves. According to the manufacturer, the dispersion parameter of the gain medium is $29.6 \text{ ps nm}^{-1} \text{ km}^{-1}$, while the group-velocity dispersion (GVD) is about $-0.056 \text{ ps}^2 \text{ m}^{-1}$. The SMF's dispersion parameter was determined to be $33.6 \text{ ps nm}^{-1} \text{ km}^{-1}$, whereas the GVD was $-0.064 \text{ ps}^2 \text{ m}^{-1}$. Hence, the equation, $D_{\text{net}} = GVD_{\text{SMF}} \times \text{Length}_{\text{SMF}} + GVD_{\text{THDF}} \times \text{Length}_{\text{THDF}}$ can then be used to obtain the net cavity dispersion, D_{net} , which is -0.89 ps^2 , indicating that the dispersion of the cavity is anomalous.

Results and discussion

After some adjustments to the PC, pulsed operation began at pump power of 308 mW. Figure 8 depicts the mode-locked optical spectrum obtained with a Yokogawa AQ6375B optical spectrum analyzer (OSA). The output wavelength was 1896.7 nm, while the 3-dB bandwidth was 2.7 nm. The dips in figure 8 are due to moisture, as they match the water absorption lines. The spikes on both sides of the optical spectrum are Kelly's sidebands. Their presence is proof that the laser is operating in the anomalous dispersion regime. The mechanism behind these sidebands is a periodic interference between the soliton pulse and the dispersive waves within the cavity. When the pulse circulates in the cavity, it experiences periodic perturbations from the various components, causing it to emit a small amount of low-intensity radiation called dispersive waves. While most of the dispersive waves interfere destructively and disappear, when the phase difference between the dispersive wave and soliton pulse is a multiple of 2π after one full round trip, constructive interference occurs, leading to the formation of Kelly's sidebands.

The working mechanism of the Fe-BTC SA to modulate pulses is based on Pauli's blocking effect. When the Fe-BTC surface is exposed to photon energy that exceeds the material's bandgap, the electrons in the valence band absorb the incident energy and are excited to the conduction band. In a low-intensity regime, most incident photons are absorbed, resulting in low initial transmission and high absorption. When the electron is excited, the generated photoexcited carriers rapidly establish a Fermi–Dirac distribution. These photoexcited carriers then cool via intraband scattering, followed by electron–hole recombination, until the hole distribution reaches equilibrium. Under high-intensity excitation, where the pump intensity is increased, the density of

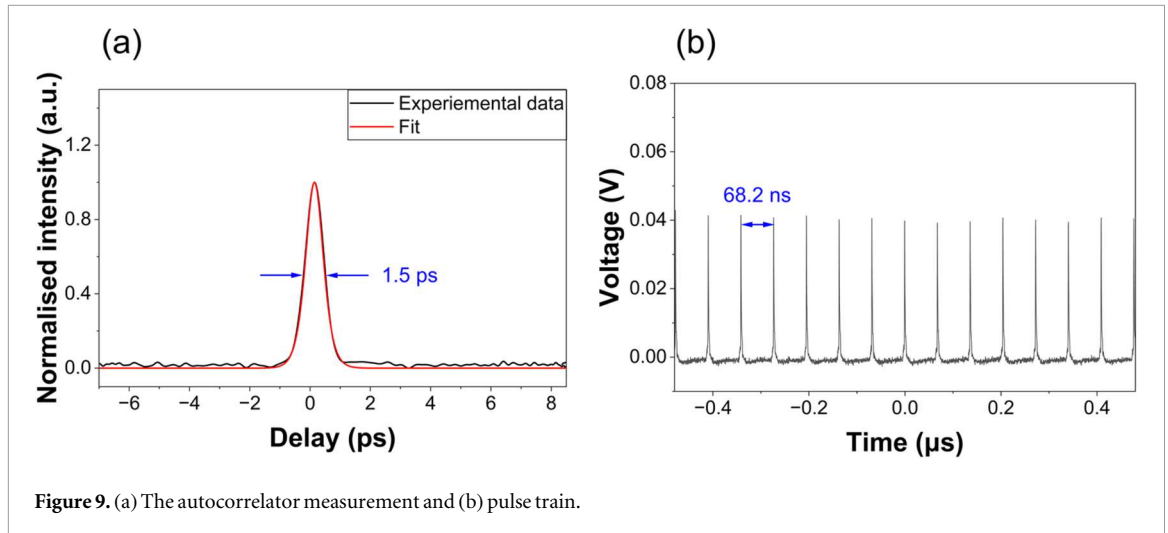


Figure 9. (a) The autocorrelator measurement and (b) pulse train.

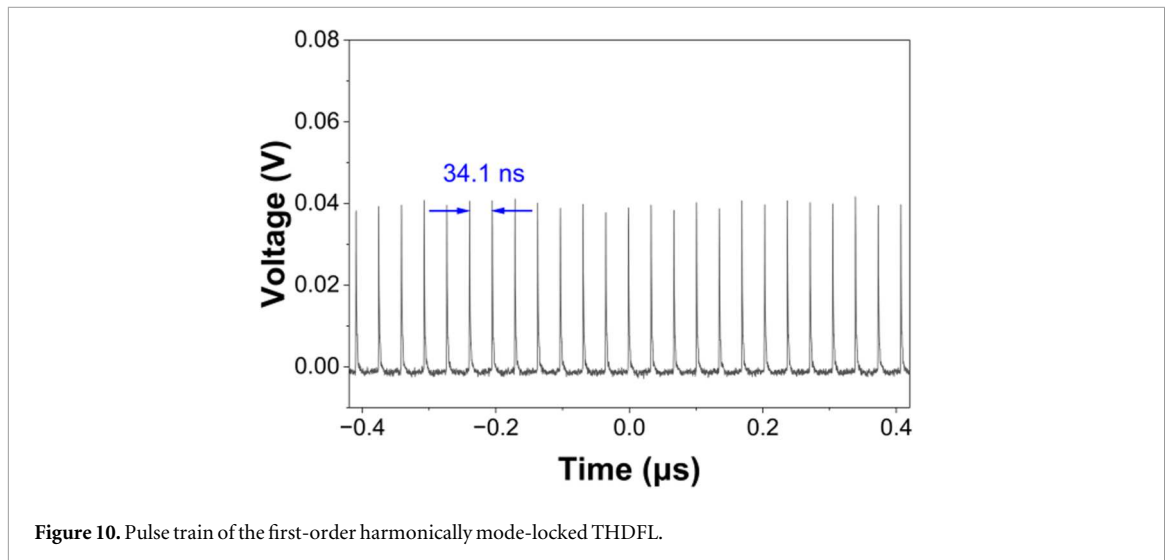


Figure 10. Pulse train of the first-order harmonically mode-locked THDFL.

photogenerated carriers increases substantially. As a result, the available states at the edge of the conduction and valence bands become fully occupied. According to the Pauli Exclusion Principle, no two identical electrons can occupy the same quantum state. Further absorption of incident photons is blocked, a phenomenon also known as Pauli blocking [47]. It leads to optical bleaching, in which the Fe-BTC becomes transparent to incident light, resulting in the high-transmission state (saturable absorption) required to facilitate mode-locking and pulse formation.

The pulse width profile was characterized with an APE PulseCheck autocorrelator, yielding a value of 1.5 ps from a sech^2 fit. It is shown in figure 9(a). Using the 3-dB bandwidth and the pulse width, the calculated time-bandwidth product (TBP) was approximately 0.337, indicating chirping, slightly higher than the transform limit of 0.315. The pulse train shown in figure 9(b) was measured with a fast photodetector and a Rohde & Schwarz RTM3004 oscilloscope. It has a measured repetition frequency of 14.7 MHz, which corresponds with the equation $f = c/nL$, where L is the length of the overall cavity, c is the speed of light in a vacuum, and n is the fibre core's refractive index.

When the pump power exceeds 450 mW, pulse splitting occurs, and the system exhibits first-order harmonic mode-locking, as shown in figure 10. Harmonic mode-locking is where the system operates in the mode-locking regime, but the repetition rate of the pulse is an integer multiple of the fundamental repetition rate, depending on the order of the harmonic. This transition from the fundamental to the harmonic mode-locking regime is attributed to soliton energy quantisation and the subsequent repulsive interactions between multiple pulses [48]. The pulse interval in figure 10 is about 34.1 ns, which is half of the pulse interval in the fundamental mode-locking regime. The corresponding repetition rate of 29.4 MHz is two times the fundamental repetition rate. Higher-order harmonic mode-locking can be achieved with higher pump powers.

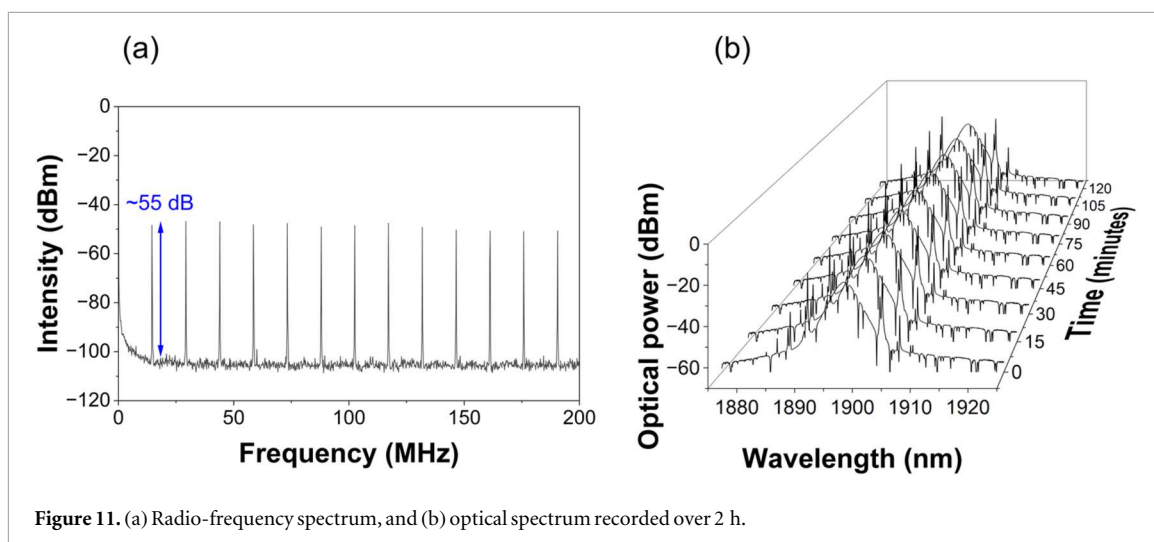


Figure 11. (a) Radio-frequency spectrum, and (b) optical spectrum recorded over 2 h.

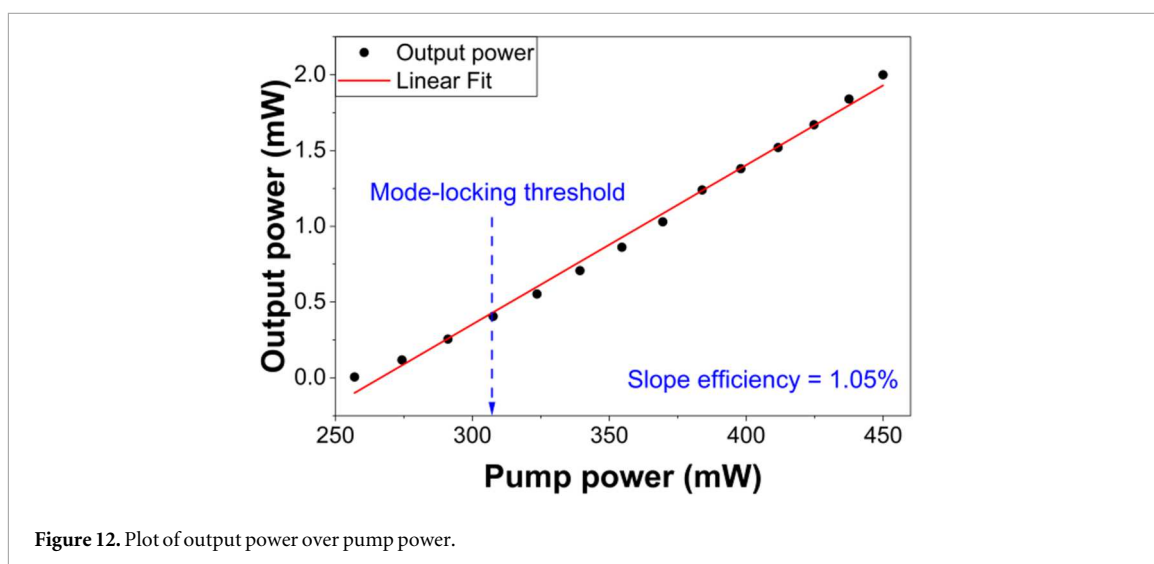


Figure 12. Plot of output power over pump power.

A Rohde & Schwarz FPC1000 radio-frequency spectrum analyzer and a photodetector were used to analyze the pulse, and the results are shown in figure 11(a). The fundamental peak at 14.7 MHz confirms the oscilloscope-measured repetition frequency. The SNR was about 55 dB, indicating that the THDFL operated stably. The system's stability was further tested by recording the optical spectrum every 15 min for 2 h. As shown in figure 11(b), the spectrum remained largely unchanged throughout the experiment, with only a slight shift in the centre wavelength of less than 0.2 nm. The experiment was successfully repeated three months after the original. It showed the reliability of the Fe-BTC SA and that the results are repeatable.

The plot of output power versus pump power is shown in figure 12, with a maximum pump power of 450 mW. An average output power of 2.0 mW with a slope efficiency of 1.05% was observed at maximum pump power. It translates to a peak power of 91 W and a pulse energy of 136 pJ. The low efficiency of the THDFL system can be attributed to two factors. Firstly, the non-saturable loss of the SA was 76.3%, which is rather high. The high intracavity loss would undoubtedly result in lower output power. Secondly, the cavity was optimized for stable mode-locking rather than power extraction. The 90/10 optical coupler delivers high intracavity power but reduces the system's output power. To enhance the laser's efficiency, the design or fabrication of the arc-shaped fibre can be improved to reduce the non-saturable loss of the SA. Another way to increase the output power is to use an optical coupler with a higher output coupling ratio, such as 80/20 or 70/30. However, it will come at a cost of lower intracavity power.

Table 1 provides a comparative summary of various material-based SAs utilized in the 2 μm region. An investigation on the broadband capabilities of graphene in mode-locking was conducted by Lau and Zulkifli [49]. Mode-locked operation was achieved in both 1563.5 nm and 1931.9 nm. The 1931.9 nm output yielded an average power of 3.4 mW, a pulse width of 1.77 ps, a peak power of 149 W, and an SNR of 65.42 dB. The SA's modulation depth was 2.13%.

Table 1. A comparison of various material-based SAs in recent years.

Type of material	Center wave-length (nm)	Pulse width (ps)	Peak power (W)	Modulation depth (%)	Average power (mW)	SNR (dB)	References
Graphene	1931.9	1.77	149	2.13	3.4	65.42	[49]
SWCNT	1913.6	0.81	—	~6.9	~6.5	~80	[50]
MoS ₂ -PVA	1927	1.51	—	12.5	<9	55	[51]
TiO ₂ -Nb ₂ CT _x	1901.4	1.64	—	19.5	<3	69.3	[52]
Fe-BTC	1896.7	1.5	91	2.1	2.0	~55	This work

In contrast, Chu *et al* [50] employed single-walled carbon nanotubes (SWCNTs) to achieve a 0.81 ps pulse with a high SNR of about 80 dB, highlighting the system's stability. The modulation depth of the SA was 3.9%, and the center wavelength of the generated pulse was 1913.6 nm, while the average output power of the system was about 6.5 mW.

A transition metal dichalcogenides-based material, few-layer MoS₂-polyvinyl alcohol (MoS₂-PVA) film SA successfully generated fundamental and third-order harmonic mode-locked pulses at 1927 nm [51]. The average power was less than 9 mW, and the pulse width was 1.51 ps. The modulation depth and SNR were 12.5% and 55 dB, respectively. A previous study reported the use of a MXene composite, TiO₂-Nb₂CT_x, as an SA for a thulium-holmium-doped mode-locked fiber laser at 1901.4 nm [52]. Paraffin oil was coated to improve the overall performance of the SA, including modulation depth, SNR, pulse width, and operational stability. The modulation depth of the SA was 19.5% while the SNR was 69.3 dB. The average power was less than 3 mW, and the pulse width was 1.64 ps.

In comparison, the Fe-BTC SA presented in this work demonstrates a competitive pulse width of 1.5 ps at 1896.7 nm. Although its modulation depth was low at 2.1%, the shorter pulse width compared to graphene, MoS₂-PVA, and TiO₂-Nb₂CT_x underscores the potential of this MOF-based material for mode-locked pulse generation in the 2 μ m region.

Conclusion

In this work, Fe-BTC is proven to be an effective SA for mode-locking in a THDFL. The SA is formed by drop-casting the MOF material onto an arc-shaped fiber. The THDFL cavity with the SA generates a stable pulse train centred at 1896.7 nm, with a pulse width of 1.5 ps and a repetition rate of 14.7 MHz. The output is highly stable with an SNR of 55 dB and a peak power of 91 W and a pulse energy of 136 pJ. The Fe-BTC-based SA demonstrates the potential of MOF materials as mode-locking elements in fibre lasers.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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