

Research Article

Thiol Functionalization of Cellulose Fabric: Synthesis and Characterization of a Heterogeneous Acid Catalyst

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Abstract

The grafting of thiol groups onto cellulosic materials represents a promising route for the development of bio-based, environmentally friendly heterogeneous acid catalysts. In this study, regenerated cellulose fabric was chemically modified through a two-step functionalization process involving preactivation and covalent grafting of -SH groups using thioglycolic acid (TGA), in the presence of p-toluenesulfonic acid (p-TsOH) as a catalyst and toluene as solvent. A parametric study was conducted to optimize reaction conditions by varying temperature, reagent concentrations, reaction time, and solvent volume. Optimal conditions were determined as follows: 0.3 equiv. of p-TsOH, 0.7 equiv. of TGA, 20 mL of toluene, at 80°C for 20 hours. Under these conditions, the grafting ratio reached 0.15, corresponding to one glucose monomer functionalized for every seven units, on average. FT-IR analysis confirmed the successful incorporation of thiol groups onto the modified matrix. These results demonstrate the potential of this material as a heterogeneous acid catalyst for applications in green chemistry.

Keywords

Functionalized Cellulose Fabric, Thiol, Thioglycolic Acid, Heterogeneous Catalyst, Green Chemistry, Covalent Grafting

1. Introduction

The transition toward more sustainable and environmentally friendly catalytic processes has driven the development of new catalytic materials derived from renewable resources. Among them, cellulose—the most abundant natural polymer on Earth—stands out as an attractive support due to its biodegra-

dability, fibrillar hierarchical structure, abundance of hydroxyl groups, and high specific surface area that can be chemically tailored [1, 2]. In this context, the chemical functionalization of cellulose represents a promising strategy for the design of bio-based heterogeneous catalysts [3].

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Received: 19 July 2025; **Accepted:** 29 July 2025; **Published:** 18 August 2025



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Among various modification approaches, the grafting of thiol (-SH) groups onto cellulose has garnered increasing interest. Thiol groups exhibit strong affinity for heavy metals, reactive surfaces, and electrophilic functionalities, making them suitable for catalytic and selective adsorption applications [4]. The introduction of thiol groups onto a cellulosic substrate is typically achieved using precursors such as thioglycolic acid, which can form ester or amide linkages with cellulose hydroxyls in the presence of acid catalysts such as p-toluenesulfonic acid (p-TsOH) [5].

Recent studies have shown that the development of acid heterogeneous catalysts based on functionalized cellulosic supports significantly enhances selectivity and reusability in reactions such as esterification, alkylation, and acid hydrolysis [6]. These catalysts offer eco-compatible alternatives to traditional homogeneous systems, which are often corrosive and difficult to recover. However, successful thiol functionalization strongly depends on key operational parameters: temperature, reaction time, thiol ligand concentration, and catalyst quantity [7]. Rigorous optimization is therefore essential to achieve a satisfactory grafting yield, thermal stability of the support, and preservation of the cellulose fabric's structural integrity [8].

In this context, the present study aims to synthesize a heterogeneous acid catalyst based on cellulose fabric modified with thioglycolic acid, by optimizing thiol grafting conditions and characterizing the resulting material using FT-IR spectroscopy. This work is part of a broader effort to valorize functionalized bio-based materials for applications in green

and sustainable chemistry.

2. Materials and Methods

2.1. Materials

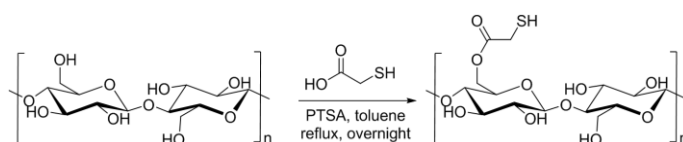
All commercial solvents and reagents were used as received from Sigma-Aldrich, Fisher Scientific Ltd., and Alfa Aesar. White primary tufting cellulose fabric with a basis weight of approximately 100g/m² was used as the cellulosic matrix. FT-IR spectra were recorded using a Bruker Tensor 27 spectrometer equipped with ATR (Attenuated Total Reflectance) technology. Elemental analyses were carried out using a Thermo Fisher Scientific Flash 2000 CHNS organic elemental analyzer.

2.2. Methods

2.2.1. Pretreatment Procedure of Cellulose Fabric (Cell)

Five pieces of white primary tufting cellulose fabric (approximately 750 mg) were dispersed in 250 mL of freshly prepared 10% (w/w) aqueous NaOH solution. The mixture was stirred for 24 hours on an orbital shaker. The cellulose fabric samples were then washed six times with 50 mL of ethanol and stored in ethanol until further use.

2.2.2. Functionalization of Cellulose Fabric with Thioglycolic Acid (Cell-ATH)



Scheme 1. Reaction scheme of cellulose fabric functionalization.

A piece of cellulose fabric (150 mg, 0.93 mmol) was immersed in dry toluene (20 mL) and treated with thioglycolic acid (257 mg, 194 μ L, 2.79 mmol) and p-toluenesulfonic acid (p-TsOH, 20 mg) as a catalyst (Scheme 1). The mixture was heated under reflux and stirred overnight at 80°C under a nitrogen atmosphere on an orbital shaker. The resulting Cell-ATH material was successively sonicated with 20 mL of acetone and dichloromethane, then dried under vacuum.

3. Results and Discussion

3.1. Functionalization of Cellulose Fabric with Thioglycolic Acid (Cell-ATH)

The functionalization of cellulose fabric through thiol

group grafting is a crucial step in the design of bio-based catalysts. Regenerated cellulose fabric was treated in anhydrous toluene, a non-polar solvent that limits moisture and activates hydroxyl groups. The grafting reaction, carried out using thioglycolic acid in the presence of p-TsOH, is based on esterification between the carboxylic acid groups of TGA and the hydroxyls of cellulose [9]. Initially, the p-TsOH catalyst protonates the carboxylic acid group of thioglycolic acid, enhancing its electrophilicity. This activation facilitates a nucleophilic attack by the hydroxyl group of cellulose on the activated carbon of the carboxylic acid, leading to the formation of an ester bond and the release of a water molecule. The reaction proceeds under mild, anhydrous conditions, which help prevent thermal degradation of the cellulose and minimize oxidation of thiol groups.

Indeed, the non-aqueous environment (dry toluene) com-

bined with an inert atmosphere prevents the oxidation of -SH groups into disulfides (-S-S-), thereby preserving their functionality in the final product [10]. This type of chemical grafting is influenced by several parameters, including temperature, solvent nature, and catalyst concentration, as shown in the literature [11, 12].

The final Cell-ATh material, enriched with accessible and stable thiol groups, demonstrates properties characteristic of heterogeneous acid catalysts. It thus represents a promising functionalized support for catalytic applications in green chemistry, particularly in esterification, condensation, or selective oxidation reactions, as reported in several recent studies [12, 13].

3.2. Optimization of the Synthesis of the Heterogeneous Acid Catalyst

Table 1. Optimization of the synthesis parameters for the thiol-functionalized cellulose catalyst (Cell-ATh).

Entry	p-TsOH (eq)	Thioglycolic acid (eq)	Toluene (mL)	Temperature (°C)	Time (h)	% S (w/w)
1	0.1	0.1	20	60	20	1.47
2	0.1	0.1	20	80	20	1.72
3 ^b	0.1	0.1	20	100	20	1.94
4	0.1	0.2	20	80	20	2.58
5	0.1	0.3	20	80	20	2.66
6	0.1	0.4	20	80	20	2.87
7	0.1	0.5	20	80	20	3.05
8	0.1	0.6	20	80	20	3.22
9	0.1	0.7	20	80	20	3.56
10	0.1	0.8	20	80	20	3.60
11	0.2	0.5	20	80	20	4.49
12	0.2	0.6	20	80	20	4.72
13	0.2	0.7	20	80	20	4.87
14	0.3	0.6	20	80	20	5.24
15 ^a	0.3	0.7	20	80	20	5.75
16	0.3	0.8	20	80	20	5.88
17	0.3	0.9	20	80	20	6.07
18	0.4	0.6	20	80	20	6.21
19	0.4	0.7	20	80	20	6.54
20	0.4	0.8	20	80	20	6.65
21	0.3	0.7	20	80	24	6.39
22 ^b	0.3	0.7	20	80	40	6.82
23	0.3	0.7	20	80	16	5.01
24 ^c	0.0	0.7	20	80	20	0.27
25 ^b	0.3	0.7	10	80	20	5.63
26	0.3	0.7	30	80	20	5.54

a: Optimal reaction conditions: Cell (0.93 mmol) and thioglycolic acid (0.7 eq) were stirred on an orbital shaker in DMF (20 mL) at 80°C for 20 hours in the presence of p-TsOH (0.3 eq). b: For entries 3, 22, and 25, the Cell-ATh material was physically damaged (degraded). c: Entry 24 corresponds to the control experiment (blank) performed without catalyst. Note.: The sulfur content (%S), corresponding to the grafted sulfur mass fraction, was determined by elemental analysis and represents the average of three independent experiments.

Optimizing the synthesis of the thiol-functionalized cellulose-based acid catalyst is essential to enhance process efficiency while minimizing economic and environmental costs. A rational approach was adopted through systematic trials in which key parameters were varied, including the amount of acid catalyst (p-TsOH), the concentration of thioglycolic acid, the volume of toluene solvent, the reaction temperature, and the exposure time, in order to determine the optimal operating conditions [14].

This optimization aimed to maximize the grafting rate of thiol groups while minimizing reagent waste and preserving the integrity of the cellulose matrix (preventing thermal degradation or weakening). The results (Table 1) clearly demonstrate the significant influence of these parameters on the sulfur content of the modified material, which serves as an indicator of functionalization efficiency [12].

3.2.1. Effect of Temperature

Reaction temperature plays a key role in the functionalization of polysaccharide-based materials, as it influences reactivity, reaction kinetics, and thermal stability. The results (Figure 1) show that increasing the temperature enhances the sulfur mass fraction grafted onto the cellulose, by promoting the esterification between thioglycolic acid and cellulose hydroxyl groups. This improvement is attributed to better site accessibility and increased polymer chain mobility [15]. However, temperature must be carefully controlled to avoid damage to the material. At 100 °C, although grafting yield initially increases, the fabric undergoes irreversible damage, including loss of cohesion, partial carbonization, and fiber degradation. These effects are caused by thermal sintering and decomposition of the thiol (-SH) groups, which are particularly sensitive to oxidation and heat [11, 16]. An optimal grafting temperature of 80 °C was identified, providing an ideal compromise between grafting efficiency and preservation of fabric integrity.

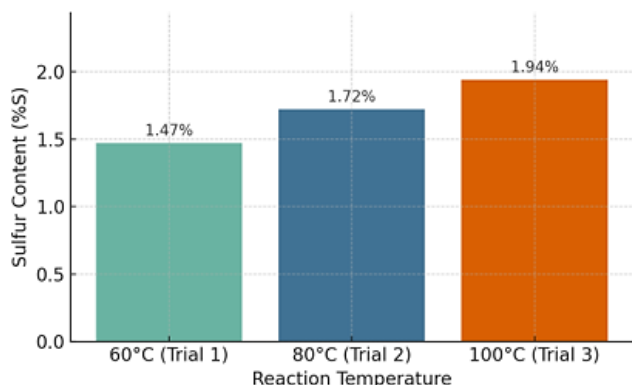


Figure 1. Effect of temperature on the sulfur mass fraction grafted onto cellulose.

3.2.2. Influence of the Catalyst p-TsOH

Para-toluenesulfonic acid (p-TsOH) plays a crucial role in the grafting of thiol groups onto cellulose. At 80 °C, increasing its concentration leads to a proportional rise in the sulfur mass fraction grafted onto the fabric (Figure 2). In the absence of a catalyst, the sulfur content remains very low, around 0.27%, indicating minimal spontaneous reactivity between thioglycolic acid and the hydroxyl groups of cellulose.

The gradual addition of p-TsOH results in a marked increase in functionalization: as soon as 0.1 equivalents are introduced, the sulfur mass fraction jumps to 3.56%. Each additional 0.1 equivalent of catalyst leads to an average increase of approximately 1% in sulfur content. A plateau in catalytic efficiency appears to be reached at 0.3 equivalents, which is identified as the optimal loading. Beyond this point, further increases yield marginal benefits and may even cause undesirable side effects, such as partial hydrolysis of the substrate or disruption of the fibrillar network [10, 11].

These results support recent findings highlighting the growing relevance of organic acid catalysts in green chemistry and the selective functionalization of biopolymers [13, 15].

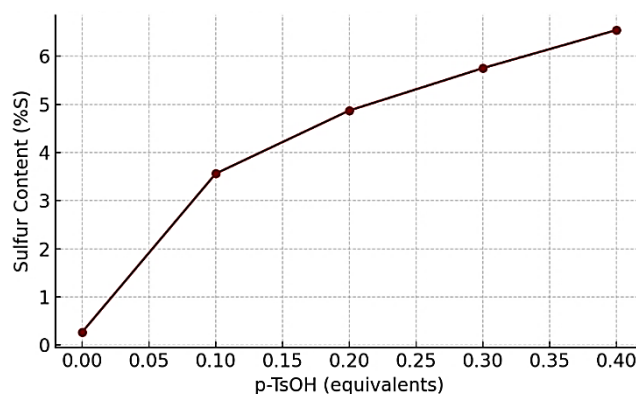


Figure 2. Effect of p-TsOH concentration on sulfur grafting efficiency.

3.2.3. Influence of the Ligand (Thioglycolic Acid)

The organic ligand used in the functionalization process has a direct impact on the overall grafting yield. Thioglycolic acid (TGA), bearing both carboxyl (-COOH) and thiol (-SH) groups, proves to be particularly reactive and well-suited to cellulose-based substrates. Experimental results (Figure 3) show a significant increase in the grafted sulfur mass fraction when the amount of TGA increases from 0.1 to 0.2 equivalents, reflecting the strong affinity of this ligand for hydroxyl groups on cellulose in the presence of an acid catalyst.

A gradual increase in grafting efficiency is then observed in the 0.3-0.7 eq range, with an approximate rise of 0.2% sulfur content per increment. Beyond 0.7 eq, the grafting curve begins to plateau, suggesting saturation of accessible reactive

sites on the support or competition between TGA molecules, possibly leading to non-productive interactions such as auto-condensation or steric hindrance [10, 15].

The efficiency of TGA lies in its ability to form covalent bonds through esterification with cellulose hydroxyl groups, and potentially through thiol-ene mechanisms with pretreated fabrics [11]. In the presence of p-TsOH, protonation of the carboxyl group increases the electrophilicity of the molecule, facilitating ester formation [16].

However, excessive TGA can lead to equipment corrosion and material degradation due to its acidic and oxidative nature. Therefore, 0.7 equivalents was identified as the optimal amount, maximizing grafting efficiency while preserving the structural integrity of the cellulose matrix [17]. Previous studies have shown that increasing the amount of precursor enhances graft interpenetration, but requires a trade-off between grafting density, chemical efficiency, and material stability [18, 19].

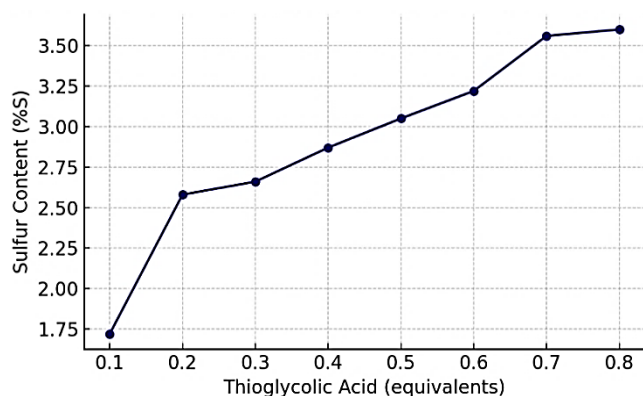


Figure 3. Effect of thioglycolic acid amount on sulfur grafting efficiency.

3.2.4. Influence of Reaction Time

Reaction time is a key parameter in the functionalization of polysaccharide supports. For the grafting of thioglycolic acid in the presence of p-TsOH, the results (Figure 4) show a significant increase in the sulfur grafted mass fraction between 16 and 20 hours. This period corresponds to a kinetically active phase, during which the majority of the hydroxyl sites available on the cellulose react with the carboxyl groups of thioglycolic acid activated by the catalyst [10, 16]. Beyond 20 hours, the grafting curve tends to plateau, indicating a gradual saturation of reactive sites or a limitation in the diffusion of reagents within the cellulose matrix. Excessive prolongation of the reaction time (up to 40 hours) results not only in marginal yield improvements but also in visible degradation of the support, due to prolonged exposure to acidic and thermal conditions that can alter the fibrillar structure of the fabric [15, 17].

An optimal reaction time of 20 hours balances grafting yield with preservation of the material's structure. This find-

ing aligns with previous studies, where extending reaction time does not necessarily improve functionalization because of limitations such as slowed diffusion, site saturation, or byproduct formation [11, 13].

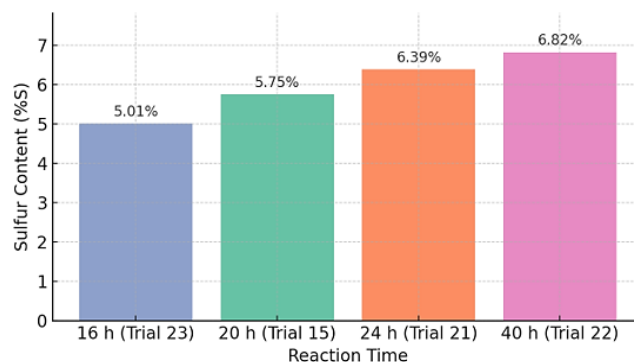


Figure 4. Influence of reaction time on the grafted sulfur mass fraction.

3.2.5. Influence of Solvent Volume

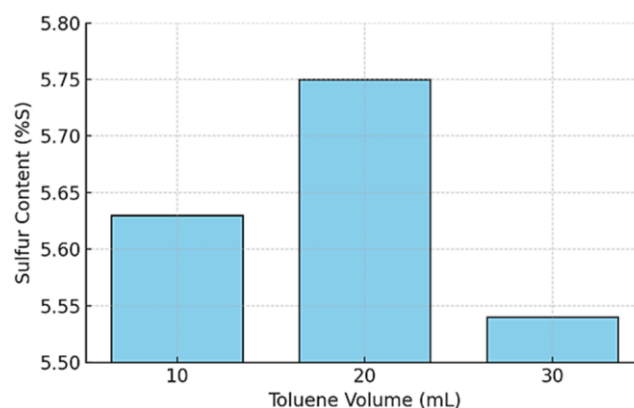


Figure 5. Effect of toluene volume on grafted sulfur content.

The evolution of sulfur content (%S) according to the volume of toluene shows a typical profile of chemical grafting optimization. An increase in sulfur content from 5.63% at 10 mL to a maximum of 5.75% at 20 mL is observed, followed by a slight decrease to 5.54% at 30 mL. It should be noted that the sulfurized fabric crumbles at 10 mL, indicating that this volume is insufficient to ensure homogeneous dispersion of the reagent in the medium. A low amount of organic solvent can lead to a locally high concentration of reagent, promoting overly localized reactions responsible for physical degradation of the cellulose matrix by chain breakage [20, 21]. Conversely, a high volume of toluene (30 mL) may cause excessive dilution of the reagent, reducing the frequency of effective collisions between thiol groups and the hydroxyl functions of the fabric. This phenomenon thus limits the grafting efficiency, despite the physical integrity of the support being

preserved [22]. A volume of 20 mL of toluene constitutes an optimal compromise, ensuring good solubilization of the reagent while preserving the stability of the fabric. This condition favors homogeneous functionalization without deterioration, in agreement with other studies on the chemical modification of cellulose supports in organic media [15, 23].

3.3. Characterization of the Modified Fabric by FT-IR Spectroscopy

Fourier-transform infrared (FT-IR) spectroscopy confirmed the covalent modifications of the cellulose fabric functionalized with thioglycolic acid (Cell-ATh). Prior to analysis, the samples were dried at moderate temperature to eliminate volatile residues. The FT-IR spectra of both native and modified fabrics (Figure 6) display the characteristic bands of the cellulose polysaccharide backbone. Two broad bands around 3400cm^{-1} and 1100cm^{-1} correspond to O-H stretching and C-O-C stretching vibrations of the glucosidic ring, which are signatures of native cellulose [1, 15].

The band at 2900cm^{-1} is attributed to C-H stretching vi-

brations of methyl and methylene groups [24], while the one at 1641cm^{-1} reflects the bending mode of weakly adsorbed water, typically present in hydrophilic amorphous regions [14].

The successful thiolation of cellulose is confirmed by the appearance of four new characteristic bands in the spectrum of the modified fabric (Figure 6). The band at 1596cm^{-1} is assigned to $\nu(\text{C}=\text{C})$ stretching vibrations, whereas the bands at 1351cm^{-1} and 1172cm^{-1} correspond to asymmetric and symmetric stretching vibrations of the thiol (-SH) group, respectively. Finally, the band at 812cm^{-1} is attributed to $\delta(\text{C-H})$ bending vibrations, indicating the presence of new covalent bonds formed during grafting [11].

The absence of these signals in the spectrum of the native fabric confirms the presence of thiol groups on the modified surface, resulting from a covalent esterification between the carboxyl groups of TGA and the hydroxyl groups of cellulose. These IR results are consistent with literature reports on the functionalization of polysaccharide-based supports for catalytic or adsorptive applications [10, 12].

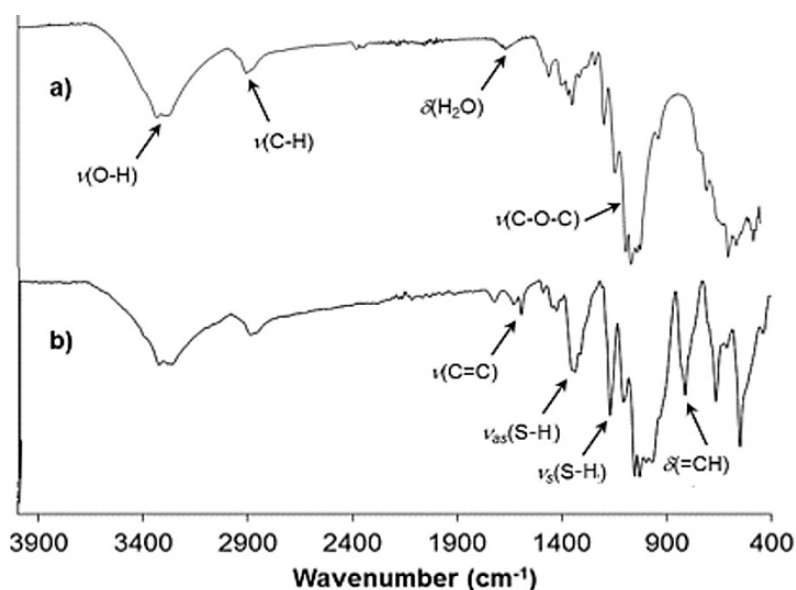


Figure 6. FT-IR spectra of (a) the virgin fabric and (b) the Cell-ATh catalyst.

3.4. Evaluation of Thiol Grafting Yield

The thiol grafting rate, calculated under optimal reaction conditions for the functionalization of cellulose with thioglycolic acid, was determined to be 0.15. This corresponds to approximately one glucose monomer out of every seven being functionalized. This grafting efficiency is significantly higher than that reported in the literature [25], who achieved a value of 0.1 on flax fibers under solvent-free conditions. These results underscore the advantage of regenerated cellulose fabric as a support material, due to its high specific surface

area, accessible reactive sites, and structural robustness. The improved yield positions this material as an ideal candidate for the synthesis of heterogeneous acid catalysts aimed at green chemistry applications.

4. Conclusion

In this study, an innovative procedure combining pretreatment and thiol-based functionalization of regenerated cellulose fabric was implemented to develop an efficient, bio-based heterogeneous acid catalyst. The covalent grafting

of thiol groups using thioglycolic acid, catalyzed by p-TsOH in an organic medium, proved both effective and selective.

The parametric study revealed the crucial influence of operational variables-temperature, reagent concentration, reaction time, and solvent volume-on the functionalization yield, as assessed by the mass fraction of grafted sulfur. The optimal conditions identified (0.3 equivalents of p-TsOH, 0.7 equivalents of TGA, 20 mL of toluene, 80 °C, 20 h) resulted in a sulfur content of 5.75%, corresponding to a grafting rate of 0.15-significantly higher than those reported in the literature for comparable systems.

Spectroscopic characterization (FT-IR) confirmed the successful introduction of thiol functionalities onto the cellulose matrix, validating the efficiency of the grafting process. This approach presents a promising platform for the design of sustainable heterogeneous catalysts, in line with the principles of green chemistry.

Future work will focus on evaluating the performance of the Cell-Ath material in key reactions such as esterification and selective oxidation, as well as investigating its stability, reusability, and integration into eco-friendly catalytic processes.

Abbreviations

Cell	Cellulose Fabric
Cell-Ath	Cellulose Fabric with Thioglycolic Acid
FT-IR	Fourier-Transform Infrared
TGA	Thioglycolic Acid
ATR	Attenuated Total Reflectance
p-TsOH	p-Toluenesulfonic Acid

Conflicts of Interest

The authors declare no conflicts of interest.

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