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Date estimation of fabrication and repair of Color garments encouragement banner

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Abstract

The Color Garments Encouragement Banner was designated a Korean Heritage in 2014 to recognize it as the most significant object of the color garments encouragement campaign. However, despite its significance, nothing is known about its manufacture. Therefore, this study attempted to analyze the materials of the banners to estimate when they were manufactured and repaired. The investigation of materials on the banner involved visual examination, literature review, microscopy, SEM–EDS, FT-IR, Py-GC–MS, ICP-MS, and LC–MS. The fabric, patch, and threads comprising the artifact were identified as cotton. FT-IR and Py-GC–MS confirmed that the repair patch was a woven blend of polyester and cotton yarns. EDS analysis indicated that the polyester was treated with titanium delustering. ICP-MS detected high concentrations of chromium that were not used in traditional dyeing techniques. The azo and sulfur compounds were identified by LC–MS analysis. The material layered on the grommet patch was thought to be a mixture of Pb, Ti with $CaCO_3$ and $BaSO_4$. Based on the overall results, the production date of the banner was narrowed down to the late 1920s, and the repair date to the mid-1950s. Although the materials used could not be identified owing to the limitations of the applicable analysis. Nonetheless, it is hoped that the analyses conducted in this study can serve as a scientific foundation for dating modern cultural heritage objects with limited handed-down record and historical documentation.

Keywords: Color Garments Encouragement Banner, Campaigns to wear color garments, Polyester, Delustering, Chromium complex, Synthetic dye, Grommet, Modern transitional period

Introduction

Korea opened treaty ports to foreign trade included western world in 1876, under the rule of the *Joseon* dynasty (1392–1910), resulting in the coexistence of the traditional Korean culture and modern western influences in all aspects of daily life, including trade, politics, diplomacy, industry, and commerce.

Some examples of this coexistence are the *Hanbok* and European-style tailored suit, *Gat* and Top hat, *Goreum,* and Button (Hong et al. 2011; Kim, 2021). This transformation led to the import of finished products as well as contributed to the production of traditional handicrafts using man-made fibers, synthetic dyes, and rubber.

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The products introduced with modernization were mass-produced industrially, unlike the traditional small-scale handicrafts. The materials used to manufacture these products quickly spread globally, providing producers worldwide with a wider range of alternatives. Thus, to examine how transition to modern period influenced cultural heritage, paying attention to the circumstances and reasons for substitution via material analysis has become imperative, rather than estimating the place of production. The Color Garments Encouragement Banner is a relic that reflects the social context of the time when modern culture was introduced. The banner features the Chinese character "色服 (*Saek Bok*; 色 means color and 服 means garment)," which means "colored garment," dyed on a black background. Traditionally, Koreans mostly wore undyed clothes; however, with the influx of new cultural influences after the opening of the port, white garments symbolized traditionality and ethnicity, whereas colored garments symbolized modernization (Choi & Hong, 2019). Wearing colored garments was advocated only by some Korean intellectuals until the 1920s and did not attract much attention. However, in the 1930s, as part of Japanese imperialist policy, campaigns to wear colored garments were led by the government throughout the country (Cho, 2010). To increase the use of colored clothing, the government trained professionals and laymen on using synthetic dyes and textile printing nationwide. In some areas, people dressed in white were banned from entering government offices (In *Yeongcheon*[永川], locals are encouraged to wear color garment, 1932). This also encouraged the industry to adopt synthetic dyes and dyeing techniques, producing more accessible colored clothing (Kim, 2018).

The Color Garments Encouragement Banner was designated a Registered Heritage of Korea in 2014 for being the only surviving artifact related to campaigns to wear color garments (Cultural Heritage Administration, 2006). However, although detailed history and provenance are unavailable, it is believed to be advertising material from the 1930s that advocated the policy of "transitioning to wearing colored clothes (Cultural Heritage Administration, 2015).

The construction of this banner is different from the conventional banners produced during the *Joseon* dynasty, such as the way it is hung on a flagpole (Takami & Eastop, 2002). Banners similar to the Color Garments Encouragement Banner were hung at the entrances of several stores in *Myeongdong*, which has been a bustling shopping district since the Japanese colonial period (Yu & Lim, 2011). The existence of a flag shop that produced and sold flags and banners since the 1920s has been confirmed (Banner and Flag Store[旗店] newspaper ad., 1924; Banner and Flag Store[旗店] newspaper ad., 1929). Another similar banner artifact from the 1930s was also reported (e-Museum, 2023), suggesting that banners of the same configuration were professionally produced at the time.

Evidence of past mending was noted on the banner, such as a tear in the "服 (*Bok*)" that was repaired by covering the damage with an additional piece of fabric, but it is unclear when the repair was performed. The estimated period and usage suggest that, in addition to grommets and sewing machines, other modern period materials and techniques may have been employed. Accurate identification of novel materials developed in the modern era, such as man-made fibers and synthetic dyes, not only provides a basis for dating but is also required for conservation treatments and management practices (Anita, 2014; Barnett, 2007; Colom & Carrillo, 2002; Hagan & Poulin, 2022; Littlejohn

et al., 2013; Sülar & Devrim, 2019; Zmeu & Bosch-Roig, 2022). Banners produced since the twentieth century have been studied for material identification, which has contributed to our understanding of conservation and degradation processes as materials change (Carlesi et al., 2016; Lee & An, 2021; Smith et al., 2016, 2019a, 2019b; Thompson et al., 2017a, 2017b).

Modern cultural heritage, where there are limited sources for estimating the date of a collection, such as archaeological findings at a site or death records of the tomb's owner, has relied on cultural context to estimate its age. The Color Garments Encouragement Banner are also lacking in handed-down records, so we can only estimate their age based on historical events. In this study, the objective is to identify the components of banner and use them as a basis for scientific estimate of date. This study attempts to analyze the components of the banner to estimate when it was made and repaired. The analyzed data were interpreted in conjunction with literature data, such as historical newspapers and photographs. The research findings are expected to serve as a basis understanding the conservation of materials used in the banner and for reconsidering the exchange of materials and technologies since the opening of the port.

Materials and Methods

Sampling

The object of the analysis was a Color Garments Encouragement Banner designated as a national registered cultural heritage; its front and reverse sides are depicted in Fig. 1. Specimens were obtained from eight points (A–G). Using tweezer, small specimens were taken from the damaged area on the reverse side. Surface material samples from the grommet patches were collected from highly fragmented and loosened areas.

Characterization

Surface observation

The banner surface was examined with a portable stereoscopic microscope (DG-3X, Scalar, JPN). Fiber cross-sections were observed with a metallurgical microscope (DM R, Leica, DEU), and images were obtained using Leica Las software (version 4.12.0). An optical microscope (ECL IPSE Ni-E, Nikon, JPN) was used to compare the longitudinal

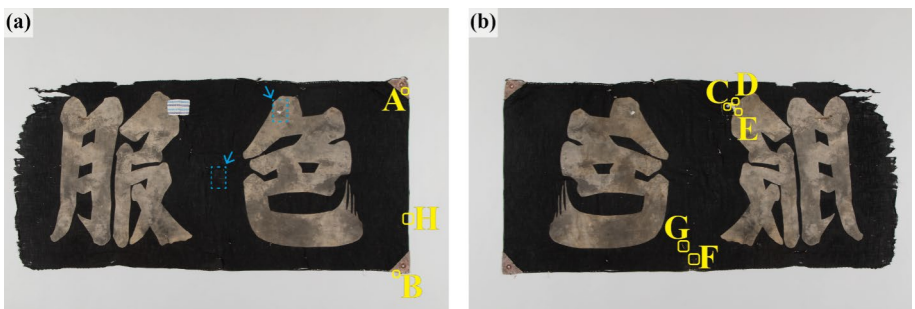


Fig. 1 Color Garments Encouragement Banner. Studied specimens were collected from marked sections; National Registered Cultural Heritage No. 615; Photo © National Women's History Exhibition Hall of Korea: **a** front side: square-shaped stitching mark remains (highlighted by blue arrow), **b** reverse side, *A* coated material of grommet patch, *B* fiber of grommet patch, *C* black fibers of ground, *D* fiber of overlay textile used for repair, *E* undyed fiber, *F* fine thread for repair, *G* heavy thread for repair, *H* thread for sewing machine. Information of samples is reported in Table S1 in Supplementary data

views of the fibers. Optical microscopic images were captured using Nikon NIS Elements imaging software (version 5.42.01).

Chemical compositions

The chemical composition was examined using a scanning electron microscope (SEM; JSM-IT300, JEOL, JPN)-energy dispersive spectrometer (EDS; X-MAXN, Oxford, UK). The sample for EDS mapping was mounted onto aluminum stubs, using carbon conductive tape and sputter coated with platinum (Pt), to avoid charging effects before the examination under high-vacuum conditions, voltage of 20 kV and 10.0 mm working distance.

The chemical compositions of the specimens were also revealed using Fourier-transform infrared spectroscopy (FT-IR; Nicolet iS5, Thermo Fisher Scientific, USA) in attenuated total reflection (ATR) mode. Fiber samples requiring isolation were defibrillated with approximately 50 μℓ of deionized water and used for analysis. FT-IR was measured with a diamond ATR crystal with a spectral range of 400–4000 cm^{-1}, a resolution of 4 cm^{-1}, and 64 scans. FT-IR spectra were collected and processed using the OMNIC software (version 9.9.473, Thermo Fisher Scientific).

Fiber identification results were confirmed using a gas chromatography-mass spectrometer (GC–MS; 8890 GC- 5977B MSD, Agilent, USA) equipped with a multi-shot pyrolyzer (Py; EGA/PY-3030D, Frontier Laboratories, JPN). Py-GC–MS can identify most fibers with trace amounts of sample (Nacci et al., 2022; Sabatini et al., 2018). Approximately 0.15 mg of polyester (Dongyang sewing thread Co., KOR) was adopted as a reference, and 0.16 mg of Sample A was placed into a pyrolysis cup. Samples were pyrolyzed at 600 °C for 1 min. The chromatographic separation was performed with a UA5-50 M (30 m × 0.25 mm × 0.25 μm). The initial temperature of 40 °C was maintained for 5 min and then increased to 310 °C at 10 °C/min, maintained for 20 min. The transfer line temperature was maintained at 280 °C. The MS ion source temperature was 230 °C and the MS quadrupole temperature was 150 °C, scanning in the 40–600 m/z range. The injection operated with a 20:1 split ratio. The pyrolysis products were eluted in constant flow mode at 1 ml/min and Helium was adopted as carrier gas. The total time of GC–MS analysis was 52 min. Data were collected and interpreted using ChemStation Software (Agilent, USA), and compound identification was performed based on mass spectra matching in the reference standard NIST17 library in the F-search system (Frontier Laboratories, JPN).

Inductively coupled plasma mass spectrometry (ICP-MS; NexION2000, PerkinElmer, USA) was used to analyze trace amounts of inorganic compounds bound during fabric dyeing and finishing. Samples were prepared by pre-dissolution with 2 ml of HNO_3 and 2 ml of H_2O, followed by dissolution by adding 2 ml of H_2O_2 and heating at 120 °C for 2 h. After dissolution, 10 ml of each sample was prepared by adding 1% HNO_3. The operating conditions are presented in Table 1.

Dyes and pigments are unique materials that can help estimate the date and location of production. Hence, analytical methods have been developed for their identification. Dye analysis of historical organic substances has been performed largely by Surface-enhanced Raman spectroscopy (SERS) (Cesaratto et al., 2018; Smith et al., 2019a, 2019b) and liquid chromatogram (Beldean-Galea et al., 2018; Güzel, 2023; Li

Table 1 ICP-MS operation condition

Parameter	Condition
RF power	1600 W
Lens voltage	9.6 V
Ar flow rate (L/min)	Plasma: 15, Auxiliary: 1.2, Nebulizer: 1.08
Scanning mode	Peak hopping
Dwelling time	50 ms
Number of replicates	3
Analytical masses	^{52}Cr, ^{56}Fe
Measurement mode and analytes	Standard mode (Cr, Fe)

Table 2 LC–MS operation condition for dyestuff identification

Instrument	Parameter	Condition
LC	Column	Acquity UPLC BEH C18 (2.1 × 100 mm, 1.7 um)
	Mobile phase	A: 0.1% formic acid in acetonitrile B: 0.1% formic acid in DW
	Flow rate	0.4 ml/min
	Gradient	Time (min) 2, 9, 13, 16 Solvent B (%) 95, 0, 0, 95
Mass	Ionization mode	ESI positive
	Scan range	80 - 1000 m/z (Full MS-ddms2)
	Collision energy	30 V
	Capillary temperature	370 °C
	Sheath gas flow rate	48 ml/min
	Aux gas flow rate	11 ml/min
	Aux gas heater temp	413 °C

et al., 2020; Petroviciu et al., 2010; Tamburini et al., 2020, 2023). As for commercial dye evaluation, liquid chromatography–mass spectrometry (LC–MS) has been proved to be a powerful technique (Terán et al., 2020). Therefore, in this study, the dyes used for black dyeing were analyzed by UPLC (UltiMate 3000 RS_UHPLC, ThermoScientific, USA) and mass spectrometry (Q-Exactive orbitrap plus, ThermoFisher Scientific, USA) from Q-Exactive plus LC–MS. The black fiber for LC–MS was a separate fragment, which presumably deteriorated and separated at the edge of the banner after a comprehensive comparison of various extraction methods, DMSO was selected for this study. The black fiber sample was heated at 80 °C in 1 ml DMSO for 24 h. The extracted sample solution was diluted 10 times with methanol solvent and used for LC analysis. Chromatographic separation was achieved on Acquity UPLC BEH C18 column (2.1 × 100 mm, 1.7 μm particle size), and the column temperature was 50 °C. The LC–MS was controlled by Xcalibur 4.0, and database searches were performed with Compound discoverer 2.3. The acquisition mass scan range for m/z was 80–1000 *m/z*. The detailed operating conditions are listed in Table 2.

Results and Discussion

Visual examination

The Color Garments Encouragement Banner depicted in Fig. 1 is a black banner with a width of 67.2 cm and a length of 138.8 cm. The fly end of the banner was partially destroyed, and there were concerns about additional damage. The background color is black, with the Chinese character "色服 (*Saek Bok*)," which was thought to be either discharge-dyed or resist-dyed on the banner. Discharge dye is a textile dye method that destroys specific parts of colored fabrics to form patterns of white or another color on a colorful background (Liu et al., 2018). Resist dye is dyed with a substance that resists dyes afterward such that only the areas not covered by the paste are affected by the dye on the colored background (Ragab et al., 2021). Metal grommets were added to both corners of the hoist end of the banner to allow for a string to be attached to the flagpole. The grommet had an outer diameter of 1.8 cm and an inner diameter of 0.8 cm. Before the grommets were inserted, triangular patches were sewn using a sewing machine. The grommet patch was sewn for extra protection and had a reddish-purple substance on its surface.

Traces of past repairs could be seen throughout the banner. Evidence of repair was observed at 19 points, including a running stitch at the edge and a stitching mark (The repair section of the banner are reported in figure S1 in Supplementary data). The same thread was used except at two points. The repairs at 15 points were made in the heavy threads, as shown in Fig. 2a. The heavy thread is a staple yarn with twists in the Z-direction that has been checked for yellowing and staining, but not dyed. The fine thread (Fig. 2b) was a staple yarn with twists in the S-direction and 4 turns/mm. Another fine thread (Fig. 2c) had different twists of 5 turns/mm and was used for sewing with a sewing machine. The banner was torn to compare the results depending on different types of damage. The tears were stitched with a running stitch, then wrapped around a seam with whip stitch. The thread was knotted at the beginning of the stitch, and at the end of the stitch, a few stitches were backstitched and tied into the stitches. The heavy thread was stitched with a single thread, while the fine thread was stitched with a double thread.

As illustrated in Fig. 3, some of the edges of the "服 (*Bok*)" were torn and repaired by covering them with a layer of separate fabric. The overlaid fabric for repair was a plain-weave fabric with a printed pattern of two different blue lines and a size of 8.0 cm × 5.6 cm. As the repair patch was only attached to the front of the banner with the heavy thread, as illustrated in Fig. 2a, the back was left exposed with a torn section, as demonstrated in Fig. 3a. Two more square stitching marks may have been repaired similarly (Fig. 1, highlighted by the blue arrow). Figure 3b presents an

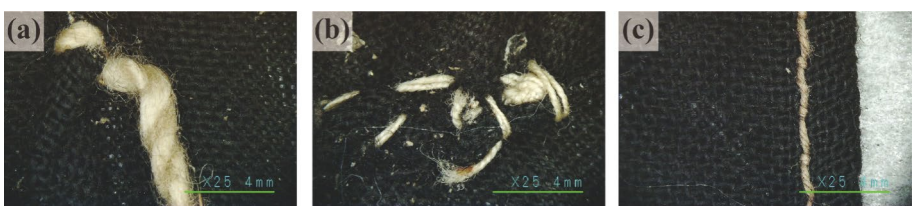


Fig. 2 Microscopic images of threads that make up Color Garments Encouragement Banner: **a** heavy thread for repair, **b** fine thread for repair, **c** thread for sewing machine. Scale bar is 4 mm, and magnification is × 25

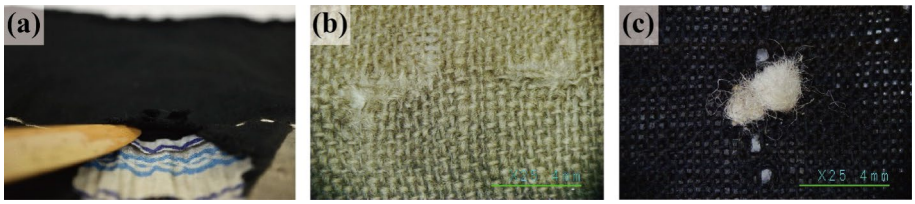


Fig. 3 Detail of Color Garments Encouragement Banner: **a** reverse side with repair patch, **b** square-shaped stitching mark, **c** remaining stitching threads. Scale bar is 4 mm, magnification is × 25

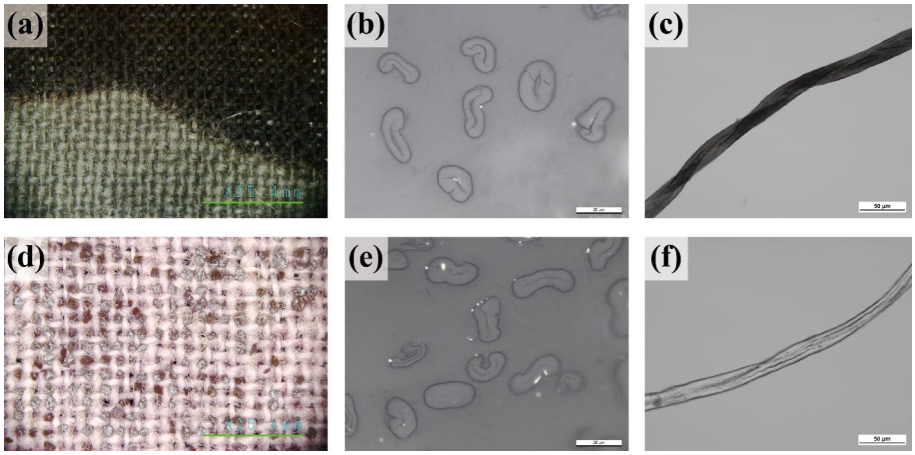


Fig. 4 Examples of fiber cross-section shapes observed via microscopy. Top row is ground textile; bottom row is grommet patch: **a** black thread of ground textile (× 25), **b** cross-section of black thread (× 1000), **c** longitudinal section of black thread (× 400), **d** grommet patch (× 25), **e** cross-section of grommet patch fiber (× 1,000), **f** longitudinal section of grommet patch fiber (× 400)

enlarged image of the sewing area. There were remnants of the sewing thread at some points, as illustrated in Fig. 3c.

Identification of fiber

Figure 4 presents cross-sectional and longitudinal images of the fiber of the banner fabric and the grommet patch. Microscopic investigation revealed that the cross-sections of the fibers were kidney-shaped, with a natural convolution resembling a twisted ribbon on the fiber axis. The average fiber staple diameter was similar to that of the black fiber (18.50 μm) and grommet patch fiber (20.27 μm). The three types of threads used for sewing and repair were also analyzed and identified as cotton threads. Some fibers with good circularity were identified in the fiber cross-section. However, this difference in circularity seem to have been due to difference in maturity and not in textile finishing. The thickness of a normal cotton fiber ranges from 12 to 22 μm. The maturity and fineness of cotton fibers are qualitative characteristics that depict considerable variability within a single variety, depending on the growing environment and development (Hussain et al., 2022; Mirza et al., 2008; Paudel et al., 2013); hence, it was impossible to identify the type of cotton fiber that made up the banner.

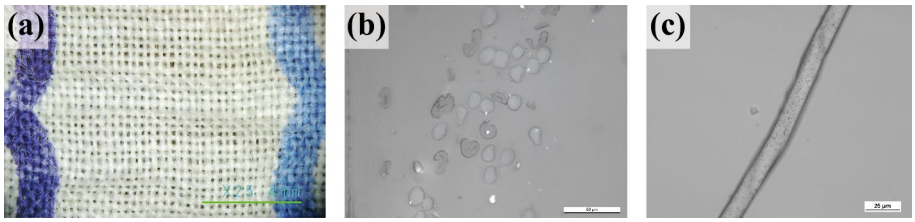


Fig. 5 Fiber properties of repair patch overlaid on damaged area: **a** weave structure of repair patch (× 25), **b** cross-section of repair patch fiber (× 500), **c** longitudinal image of repair patch after isolating from cotton fiber (× 600)

Table 3 EDS analysis of repair patch thread after isolating from cotton fiber

Sample number	Element	Mean (weight, %)[a]	SD (weight, %)[b]
Black fiber	C	75.43	0.37
	O	24.41	0.42
	Ti	0.17	0.06

[a] Mean value is an average taken at 3 different spots on the sample

[b] Standard deviation, number of spots = 3

When observing the sample from the repair patch (Fig. 5a), in addition to the kidney-shaped cross-section, another type of fiber cross-section with a round shape was identified (Fig. 5b). Black spots were also observed on the sides of the fibers with round cross-sections (Fig. 5c). To verify the type of fiber presented in Fig. 5c, EDS analysis was performed by isolating it from the cotton fiber. As indicated in Table 3, Titanium (Ti) was detected in the fibers. Ti is used as a delustering agent to control the gloss of man-made fibers with smooth surfaces and high luster (Puls et al., 2011; Rechmann, 1974). Titanium dioxide (TiO_2) has a refractive index of 2.71, which is known to have a good delustering effect because of its large refractive index (Chuang et al., 2011; Samat et al., 2016). Therefore, the spots observed on the side were estimated to result from titanium mixed in the spinning dope, reducing the man-made fiber's luster.

The fiber blended with cotton is a man-made fiber spun with a delustering agent; therefore, additional analysis was required to identify it. FT-IR spectroscopy in the attenuated total reflectance (ATR) mode is a particularly suitable method for fiber identification because it offers highly characteristic information and is fast, easy, and non-destructive (Peets et al., 2017). As demonstrated in Fig. 6, the FT-IR spectrum of the repair patch exhibits peaks corresponding to a mixture of cellulose and other fibers. To determine the composition of the blended fibers, approximately 1 cm long fibers collected from the conservation process were dissected and reanalyzed for functional groups. After isolating from the cotton fiber spectrum, the repair patch had several extra absorbance bands, most notably the peaks for C–H stretching at 2972 cm^{-1} and 2908 cm^{-1}. The aromatic overtones/combinations area is between 2500 and 1800 cm^{-1}—these bands are characteristic to polyester fiber (Junlong et al., 2012; Pilleriin et al., 2019). The FT-IR spectrum of acetate is similar to that of polyester, and the spectroscopic techniques struggle to discriminate cellulose-based fibers, preventing the exhaustive characterization of the samples (Nacci et al., 2022).

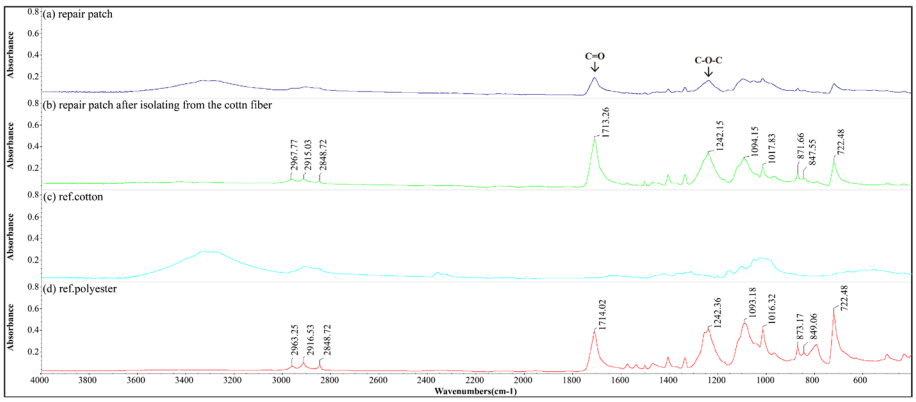


Fig. 6 ATR FT-IR spectrum of **a** repair patch, **b** repair patch after isolating from cotton fiber, **c** reference cotton fiber, and **d** reference polyester fiber

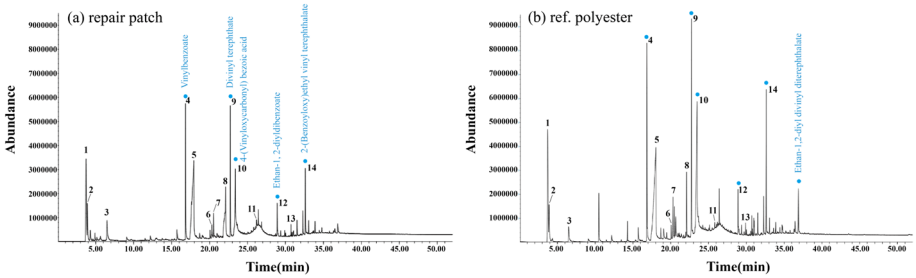


Fig. 7 Py-GC/MS chromatogram of **a** repair patch, **b** reference polyester fiber

Additional Py–GC–MS analysis was performed to clarify the characterization of the repair patch fibers blended with cotton.

Figure 7 presents Py–GC–MS chromatograms of polyester (PET; polyethylene terephthalate) reference and repair patch fiber samples. In the repair patch's chromatogram, the acetate's characteristic ion was not identified, and the pattern was most similar to that of polyester. Fourteen compounds were observed to be common in the repair patch and polyester reference sample, including 2-(Benzoyloxy)ethyl vinyl terephthalate at 32.6 min. The compounds with characteristic ions are reported in Table 4. In particular, pyrolysis markers identifiable as polyester terephthalates, including vinylbenzoate, divinyl terephthalate, 4-(Vinyloxycarbonyl) benzoic acid, ethan-1,2-diyldibenzoate, and 2-(Benzoyloxy)ethyl vinyl terephthalate, were commonly detected in the repair patch sample. Therefore, the repair patch is considered to be a fabric that is a blend of cotton and polyester.

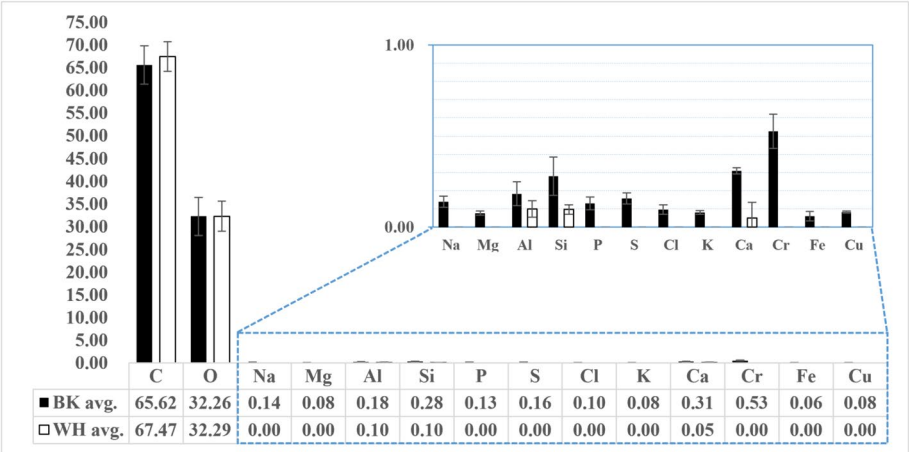
Dyestuff and mordant analysis

The Color Garments Encouragement Banner has two parts: black and other colors. Identifying the dyestuff used in historical textiles remains a challenge; however, inferring the dyestuff from the metallic mordant is possible. According to the *Joseon* dynasty literature, before the introduction of novel dyeing techniques involving the use of synthetic dyes, iron mordants were essential for black dyeing using natural dye

Table 4 Compounds of labeled peaks in Py-GC–MS chromatograms of repair patch sample, reference fiber

No. of peak	Retention time (min)		Compound (matched)	Characteristic ions (*m/z*)
	Repair patch	Ref. polyester		
1	3.724	3.722	Carbon dioxide*	44
2	3.900	3.906	Acetaldehyde*	29
3	6.491	6.484	Benzene	78
4	16.836	16.836	Vinylbenzoate	51, 105, 148
5	17.915	18.022	Benzoic acid	51, 105
6	20.311	20.294	2,2-Dimethyl-1-(2-vinylphenyl)propan-1-one*	77, 131
7	20.520	20.514	Biphenyl	76, 154
8	22.103	22.099	1(3H)-Isobenzofuranone, 3,3-dimethyl-*	43, 91, 147
9	22.739	22.743	Divinyl terephthalate*	104, 175
10	23.396	23.484	4-Acetylbenzoic acid	65, 104, 149
11	26.189	25.822	*	*
12	28.905	28.905	Ethan-1,2-diyldibenzoate*	51, 105
13	30.729	29.901	Eicosane	57, 99
14	32.622	32.628	2-(Benzoyloxy)ethyl vinyl terephthalate*	297

*Cannot be identified or has a low quality of library matching (quality < 80)



	C	O	Na	Mg	Al	Si	P	S	Cl	K	Ca	Cr	Fe	Cu
■ BK avg.	65.62	32.26	0.14	0.08	0.18	0.28	0.13	0.16	0.10	0.08	0.31	0.53	0.06	0.08
□ WH avg.	67.47	32.29	0.00	0.00	0.10	0.10	0.00	0.00	0.00	0.00	0.05	0.00	0.00	0.00

Fig. 8 EDS analysis (wt%) of background textile used in Color Garments Encouragement Banner

resources (Park & Chang, 2017, 2020). Therefore, the presence or absence of iron in black-dyed fabric indicates whether the dye is natural or synthetic. EDS analysis was performed to identify the metal mordant components; the results are summarized in Fig. 8. EDS analysis revealed that the iron (Fe), copper (Cu), and chromium (Cr) in the black background fabric sample were not detected in the white letter partial fiber sample. Cr is a metal mordant not recorded in *Joseon* dynasty documents and is a common ingredient in synthetic dyestuffs. To clarify whether Fe or Cr was the main component involved in the dyeing, ICP-MS analysis was performed, and the results are presented in Table 5. The Fe component of the black fiber was 29.913 μg/g, which was detected as 1/16 of the Cr element (479.804 μg/g). Based on the amounts of Fe and Cr, it was thought that Fe was added for color development and fastness in the

Table 5 Concentration of metals (Cr, Fe) in black fiber in Color Garments Encouragement Banner

Sample	Element	Concentration (μg/g)[a]	RSD (%)[b]
Black fiber	Cr	479.804 ± 2.69	0.56
	Fe	29.913 ± 0.40	1.35

[a] Data were expressed as mean ± SD

[b] Relative standard deviation, number of samples = 3

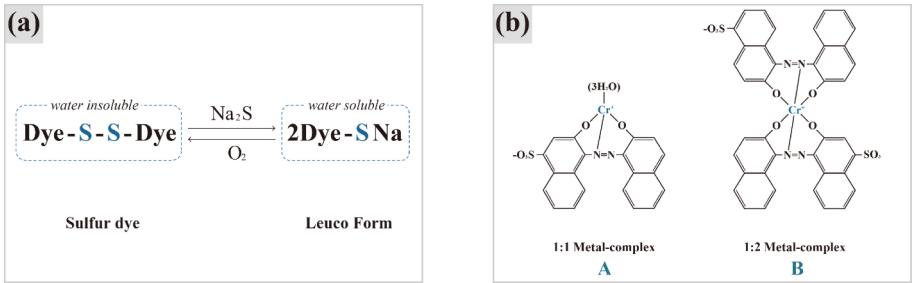


Fig. 9 Dyeing mechanisms of sulfur dye and metal-complex dye: **a** reduction–oxidation processed in sulfur dyeing, **b** examples of structures of chromium complex. A: 1:1 complex, B: 1:2 complex

process of developing a dark color and that the metal element that directly contributed to dyeing was Cr, which was detected in large amounts.

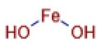
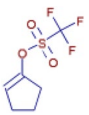
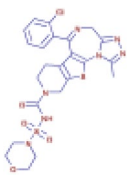
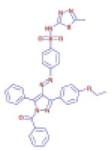
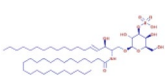
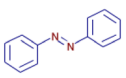
Cr is mainly used as an oxidizing agent for dyeing sulfur dyestuffs or as a mordant agent to form metal complexes. Sulfide dyes with bonds such as –SH, –S–, and –S–S– are synthetic dyes mainly used for dyeing cellulose fibers (Khattab et al., 2020). Figure 9 illustrates the oxidation–reduction reactions of the sulfide dyes. Sulfide dyes are known to be reduced in sodium sulfide (Na_2S) solutions and migrated to cellulose in the R-SNa state (Yao et al., 2015; Wang et al., 2001; Tehrani-Bagha & Holmberg, 2013). After being migrated to the fiber, the leuco dye must become water-insoluble via oxidation to terminate the dyeing process completely. Typical oxidizing agents used in this process are sodium dichromate ($Na_2Cr_2O_7$) and potassium dichromate ($K_2Cr_2O_7$), which contain Cr (Hickman, 1993; Perkins & Crew, 1975; Teli et al., 2001).

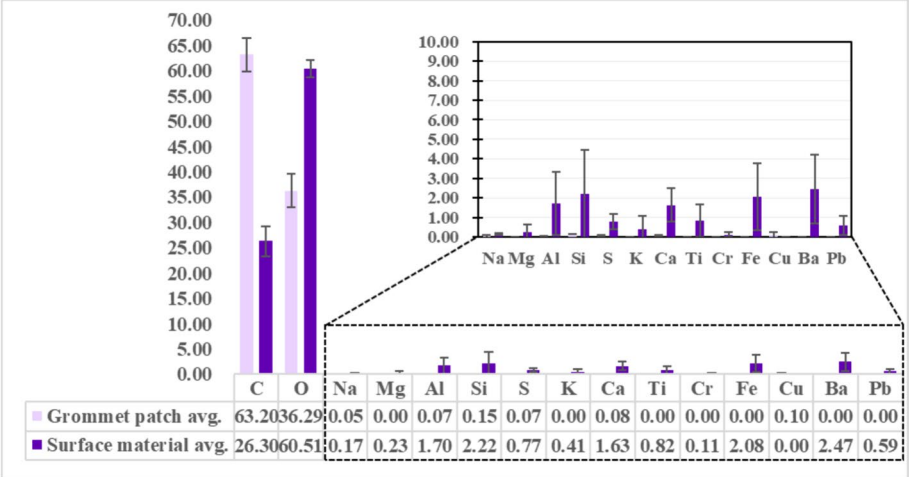
Chromium was first applied to cotton dyeing in the West in 1875 by Bird (Bird, 1875) and was primarily used as a mordant for cotton dyeing until the late nineteenth century (Benkhaya et al., 2020; Hummel, 1885). Until environmental pollution became an issue, chromium has become the dominant mordant in the synthetic dye industry and is included in most metal-complex dyes (Aksu & Karabayır, 2008; Bardole et al., 1998; Velusamy et al., 2021). Dyes with hydroxyl groups in the ortho position, as depicted in Fig. 9-b, can form metal complexes with 1:1 or 1:2 structures via metal mordanting (Beffa, 1984; Choe et al., 2016). In 1889, an acid-mordant dye that formed a Cr complex upon treatment with dichromate was commercialized (Park & Shore, 2006). Since the mid-1910s, pre-metalized direct, acid, and reactive dyestuffs have been introduced (Park & Shore, 2006; Stallmann, 1960). Several cotton fabric swatches printed with mordant dyestuffs and chromium were identified in a book published in 1902 (Farbenfabriken vorm. & Friedrich Bayer & Co, 1902).

Given the ratio of chromium to sulfur (1:3.5) detected in the banners, it is difficult to conclude whether the residual chromium-based oxidant released into the dye wastewater at the end of its role is greater than the chromium complex adsorbed on the fibers. Sulfonic acid groups also impart solubility to other dyes; therefore, the black color of the Color Garments Encouragement Banner is attributed to dyeing with chromium-complex dyes containing sulfonate groups ($–SO_3Na$, $–SO_2$, $–SO_3$) (Dodangeh et al., 2021; Yiqi & Carman, 1996).

To substantiate the existence of sulfur and azo-based components, presumed to comprise the fundamental elements of the dye in black fiber, the mass spectra of black fiber's compounds were acquired in positive mode using ESI and subsequently compared with data from the database. The compounds were identified by comparing their structures to those found in previously conducted studies. The compounds that were detected in high quantities are shown in Table 6, and correspond to the iron, sulfur, and azo groups. The Fe identified by EDS and ICP-MS analysis is 'Iron(2+) dihydroxide', which is believed

Table 6 Analysis of black fiber of the Color Garments Encouragement Banner using LC–MS

Identified compound	Structure	Retention time (min)	[M + H] (*m/z*)	[MS/MS] (*m/z*)	Chemical formula
Iron(2+) dihydroxide		0.03	99.94818	–	H_2 Fe O_2
1-Cyclopenten-1-yl trifluoromethanesulfonate		0.66	217.01442	217.01446	C_6 H_7 F_3 O_3 S
Methylsulfonylmethane		0.91	95.01652	–	C_2 H_6 O_2 S
Tetramethylene sulfoxide		1.27	105.03719	105.03730	C_4 H_8 O S
		1.48	105.03719	105.03729	
6-(2-Chlorophenyl)-1-methyl-N-(4-morpholinylsulfonyl)-7,10-dihydro-4H-pyrido[4′,3′:4,5]thieno[3,2-f][1,2,4]triazolo[4,3-a][1,4]diazepine-9(8H)-carboxamide		6.16	562.10895	–	C_{23} H_{24} Cl N_7 O_4
		6.27	562.10931		
4-[(E)-{4′-[(E)-{2,4-Diamino-5-[(E)-(4-sulfonatophenyl)diazenyl]phenyl}diazenyl]-4-biphenylyl}diazenyl]-2-hydroxybenzoate		6.26	650.16107	–	C_{33} H_{27} N_7 O_4 S_2
		6.52	635.14703		
5-Methyl-2′,3′-bis-O-(methylsulfonyl)-5′-O-trityluridine		6.42	657.15967	–	C_{31} H_{32} N_2 O_{10} S_2
3-O-Sulfogalactosylceramide		9.29	892.65570	–	C_{48} H_{93} N O_{11} S
Azobenzene		5.03	183.09186	183.09193	C_{12} H_{10} N_2



	C	O	Na	Mg	Al	Si	S	K	Ca	Ti	Cr	Fe	Cu	Ba	Pb
Grommet patch avg.	63.20	36.29	0.05	0.00	0.07	0.15	0.07	0.00	0.08	0.00	0.00	0.00	0.10	0.00	0.00
Surface material avg.	26.30	60.51	0.17	0.23	1.70	2.22	0.77	0.41	1.63	0.82	0.11	2.08	0.00	2.47	0.59

Fig. 10 EDS analysis (wt%) of grommet patch used in Color Garments Encouragement Banner

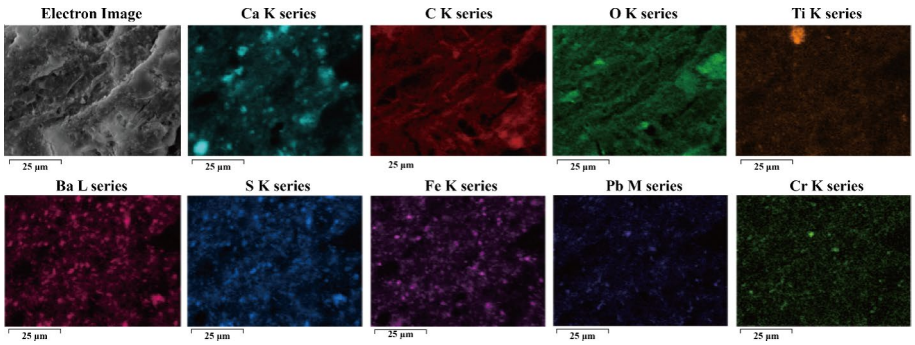


Fig. 11 Representative set of EDS images obtained for single site on surface material. Scale bar represents 25 µm

to have originated from pollutant on the oxidized banner surface. Among the identified compounds, 7 contained sulfonate groups, and azobenzene corresponding to azo groups (–N=N–).

Identification of surface substances on grommet patch

The surface of the grommet patch on the hoist end of the banner was partially covered with a layer of residual polishing substances, as illustrated in Fig. 4d. The residual substances on the surface are fragments of inconsistent sizes and thicknesses. It has a silver or reddish-purple color depending on the angle of light. EDS analysis of the exfoliated specimen of the surface substances from the grommet patch revealed that barium (Ba), calcium (Ca), Ti, and lead (Pb) were the major components, as indicated in Fig. 10, while they were seldom detected in grommet patch fibers. These materials were thought to be simply coated on the surface without chemical bonding.

SEM–EDS mapping analysis was performed to compare the composition distribution of the surface materials. The elemental distribution of the surface material obtained at a single point in one of the surface material samples analyzed by EDS is illustrated in Fig. 11. As shown in the elemental distribution map detailing the presence of each

element, the composition appearance of barium and sulfur was similar, and that of lead, chromium, and titanium was similar. The distribution of barium seen by EDS mapping suggested a lead-based ground, likely composed of lead mixed with chromium and titanium; the three elements are observed throughout the composition in EDS.

The typical extenders, calcite ($CaCO_3$) and barite ($BaSO_4$), have been utilized in Korea since the late 1920s (Kim et al., 2017). The defects of $BaSO_4$, which have low absorption and adhesion to fibers and easily fall off even during laundering, could explain the current state in which most surface substances were lost (Ko, 2007). Ti and Pb are components of representative white pigments, whereas lead white pigment ($2PbCO_3 \cdot Pb(OH)_2$) was replaced by other white pigments in the late 19th and early twentieth centuries because of problems such as lead poisoning (Barnett et al., 2006; Habashi, 2016; Rutherford et al., 1967). Therefore, the substance found on the surface was considered a part of the calcium and barium extenders.

Estimation of date of production and repair for Color Garments Encouragement Banner

The Color Garments Encouragement Banner comprised three types of plain-weave fabric: a textile background, a grommet patch, and an overlay textile used for repair. The selvage remained on both sides, indicating that a plain-woven cotton fabric with a width of 67.2 cm was used. The width of the cotton fabric woven with the traditional Korean handloom was 30–40 cm. After the advancements in the cotton industry and the introduction of improved looms such as the batten loom in the 1900s, weaving wide-width cotton fabrics became possible. Wide cotton fabrics were imported before and after the opening of the port, and with improved and modernized looms, cotton fabrics of varying widths were mass produced in Korea (Lee, 2010). In addition, two types of repair threads with different thicknesses and one type of thread utilized in sewing machines were identified. Traces of novel technology can also be found in the stitching around the edges of banners. The hoist end was completed by rolling up the seams and pressing them onto a sewing machine. The first sewing machine was introduced to Korea in 1877 (Gwan, 1928). The Singer sewing machine company had a near monopoly on the market since 1906, when it listed three separate stores in Korea. However, from the late 1930s, various brands such as Brother and Mitsubishi entered the Korean market (Godley, 2006, 2008).

In addition to the sewing machine thread, two other types of threads were utilized to repair the 19 points. Of these, 15 locations utilized a heavy thread, two utilized a fine thread, and the stitch pattern was the same; therefore, the repair time was determined to be similar. The heavy thread was utilized to stitch the repair patches. The repair patch was woven from a blended yarn composed of polyester and cotton. The first synthesis of polyester in 1942 by John R. Whinfield and James T. Dickson was conducted in the United Kingdom at the beginning of World War II, under the Calico Printers Association (Damayanti & Wu, 2021). Trial production of Terylene (polyethylene terephthalate) was initiated in 1947, large-scale production began in 1953 (Veit, 2003). The polyester fibers that comprise the repair patch indicated that Ti was used as the delustering. The artificial luster of man-made fibers has been studied earlier because it needs to be controlled for practical utilization. In 1928, a method was patented for adding titanium to man-made cellulose fibers to control their luster (United States Patent US1692372A, 1927). Since the early 1930s, TiO_2 has been adopted as a delustering agent to control the luster of man-made fibers (Periyasamy,

2023; Puls et al., 2011; Rujido-Santos et al., 2022). Considering that the cotton thread utilized to sew the repair patch was identical to the thread utilized to sew the other repaired parts, alongside the development and commercialization of polyester and TiO_2 delustering agents, it is estimated that the banner was repaired in the mid-1950s. In 1968, the Republic of Korea began weaving polyester fabrics (Lee & Hong, 2001). While nylon was introduced to Korea as a military material (Lee & Hong, 2001), it is possible that polyester was brought into the country by the USA or UN forces during the chaotic period of the Korean War (1950–1953). Campaigns to wear colored garments did not attract much attention during World War II (1939–1945). Even after independence (August 15, 1945), Korea was not completely organized until the 1950s because of the outbreak of war. Until 1955, the expression "色服 (color garment)" persisted, with state officials being advised to wear domestically made color garments to comply with "The Law for Improving Daily Life of People" (Recommendation to wear domestic color garments, 1995). Given the limited use of the term "color garment" in print media and administrative documents since the 1960s, it is possible that the banner was repaired in the mid-1950s, when the term "色服" was in use.

SEM–EDS and ICP-MS analyses confirmed the presence of Cr in the black-dyed areas. In the traditional Korean black dyeing technique, which is based on natural dye resources, Fe mordanting was essential for black coloration; therefore, the detection of high concentrations of Cr suggests the utilization of synthetic dyes. A total of 178 types of synthetic dyes were confirmed to have been distributed in Korea from the late 1890s to the 1940s (Pak, 2018). Among them, seven were direct black dyes, twelve were sulfur dyes, and five were mordant dyes. Azoic acid dyes, developed in 1912, were imported in 1940 (Pak, 2018). Although determining which of these dyes were used is difficult, these can be narrowed down to azo-based chromium complex dyes that can be used to dye cellulose fibers. The LC–MS analysis identified sulfonate groups and an azo compound, Azobenzene, in the black cotton fibers. The inability to identify the exact dye was attributed to the limited detection of Cr by LC–MS.

Metal grommets, invented in the Victorian Era (1837–1901), were also used at the corners of the hoist end and were not found in the premodern era (Finch, 1991). Metal grommets were also found on banners promoting restorative drinks produced in the 1930s (e-Museum, 2023). Grommets were inserted over a cotton patch to minimize wind damage. Ca, Ba, Ti, and Pb, which were not found in the fibers of the grommet patch, were detected on the surface substances. The main ingredients of the surface substances, Ca and Ba, are believed to be derived from the typical extenders, calcite, and barite, respectively, which have been used in Korea since the 1920s (Kim et al., 2017). Ti and Pb were the pigment components compounded therein. This indicates that paint containing extenders and pigments was presumably coated onto the surface of the patch for durability. Hence, excluding repair materials, it is plausible that the requisite materials for the fabrication of banners were conceivably procurable by the late 1920s.

Conclusions

This study attempted to analyze the materials and techniques of the Color Garments Encouragement Banner, thereby to providing a basis for estimating the time they were made and repaired and helping establish a timeline for the introduction of novel

fabrics and dyestuffs. Microscopy, SEM–EDS, FT-IR, Py-GC–MS, ICP-MS, and LC–MS analyses were employed, and the literature was also reviewed.

Scientific analysis confirms that the banner was produced by introducing novel materials and technologies, including modern looms and sewing machines, and dyestuffs formulated with Ba and Ca extenders. The LC–MS analysis did not conclusively identify the dyes compared to other studies, and limitations in identifying the Cr-complex dye by HPLC-ICP-MS or IC-ICP-MS. Nevertheless, the presence of azo and sulfonate groups, as identified by LC–MS analysis, was assumed to constitute part of the black dye component, possibly in conjunction with Cr. Barite, a presumed surface substance of grommet patches, was found to have been introduced in the late 1920s and is the latest of the constituent materials of the banner fabric. This implies that it was possible to create the banner in the late 1920s.

The timing of the repair, which was another point of investigation, could be estimated based on the repair patch. Polyester fibers blended with cotton were treated with Ti delusters, which became commercially available after the mid-1950. Therefore, the banner was estimated to have been repaired using repair patches after the mid-1950s.

The above material analysis allows us to reconsider the timing of production of the Color Garments Encouragement Banner, and its repair. The banner had a significant role in the color garments encouragement campaign, which promoted modernization, and it could be confirmed that modern technologies and novel materials were introduced in actual production. Although this study is limited by its inability to identify materials, it is significant in that it provides a basis for estimating the time of the production and repair of the banner, which has remained unclear despite the designation of the banner as a nationally registered cultural heritage.

Thus far, the dating of textile cultural heritage has commonly relied on archaeological findings from the site, the tomb owner's death record, or the prevailing cultural context. However, most modern cultural artifacts have limited records to estimate when they were manufactured. Therefore, this study is significant in that it analyzes materials and scientifically infers the fabrication dates of modern cultural heritage based on the timeline of development and importation of the identified materials. It is anticipated that the identification of materials and the establishment of a scientific database will be actively utilized in dating the production of modern cultural heritage.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s40691-024-00390-y.

Supplementary Material 1.

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Author contributions
SP carried out all the practical work and prepared the initial draft of this manuscript. BA conceived the experiments and managed NRICH's support to make this study a success. CY participated in discussions and manuscript writing, review and editing. All authors read and approved the final manuscript.

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Availability of data and materials
Data and analysis collected during the study are available from the corresponding authors upon reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Competing interests
The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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