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ORIGINAL ARTICLE

Effectiveness of X-Ray Fluorescence Spectrometer Technique in Analysis of Nano-Coated Textile Material for use in Forensic Science

[1]Parshuram Singh, [2]Sapna Balayan,[3]R K Sarin,[4]Utkarsh Jain

ABSTRACT

CONTEXT: Forensic examination is conducted to detect, identify, and investigate the crime to figure out the pieces of evidence and connect it to the perpetrator of the crime. The nanoparticles play a crucial role in the forensic analysis of the evidence obtained at the crime scene. These nanoparticles can be characterized by various scanning and X-Ray techniques. The XRF technique provides an effective analysis of elemental composition of the materials.

AIMS: This study aims to perform a differential analysis of nanomaterials of coated and non-coated samples through X-Ray fluorescence spectroscopy.

MATERIALS & METHOD: Firstly, the titanium dioxide (TiO_2) nanoparticles were synthesized by using a hydrothermal method. This nanomaterial was then characterized with distinct techniques such as Dynamic light scattering (DLS), X-Ray diffraction (XRD), and Ultraviolet-visible spectroscopy (UV-VIS). Furthermore, the TiO_2 nanoparticles were coated on the surface of rexine, paint, and glass to observe the composition of elements in nano-coated and non-coated samples. Moreover, the surfaces were characterized by using SEM, and the elemental composition was determined through XRF.

RESULTS: The results exhibit a distinctive difference in the concentration of titanium obtained in glass samples. However, the analysis on rexine and paint samples shows that the difference in the quantity of titanium is less when the nanocoated and non-coated samples were analyzed.

CONCLUSIONS: It was concluded that titanium is already present during the manufacturing of rexine and paint therefore, the nanoparticle coating of TiO2 doesn't create a large difference. Besides, there was a significant difference in nanomaterials coated and non-coated glass samples.

KEY MESSAGES: The forensic investigation can be more qualitative with nanomaterial coating on the surface of glass, fabrics, and paint samples. These materials can be easily characterized with surface and X-ray techniques to provide efficient data.

KEYWORDS | x-ray fluorescence, scanning electron microscopy, dynamic light scattering, DLS

Author's Credentials:
[1]Student, Amity Institute of Forensic Sciences, [2]Student, [4]Assistant Professor, Amity Institute of Nanotechnology, Amity University, Noida-201313, Uttar Pradesh, India, [3]Director, Andhra Pradesh Forensic Science Laboratory, Guntur, Andhra Pradesh-522502, India.

Corresponding Author:
***Utkarsh Jain**, Assistant Professor, Amity Institute of Nanotechnology, Amity University, Noida-201313, Uttar Pradesh, India*

Email:
ujain@amity.edu

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INTRODUCTION

Recently, nanotechnology has emerged as a harbinger of new technologies in product development due to the unique properties of the materials at their nanoscale. This study was conducted for the examination of different samples such as rexine, paint, and glass. Effective analysis was done for each sample coated with titanium dioxide (TiO_2) nanoparticles and non-coated samples by using the XRF technique for forensic applications.

METHOD & MATERIALS

Chemicals and Equipments

Titanium dioxide is widely used in consumer products including textile and paint materials. Therefore, titanium dioxide nanoparticles were selected to analyze the surface of these materials (rexine, paint, and glass) during forensic analyses. The rexine, paint, and glass slab were purchased from a local vendor. Titanium tetra isopropoxide (TTIP), ethanol, and nitric acid were procured from US-based Sigma-Aldrich. Distilled water (DW) was used for preparing the samples that were used in this research. The apparatus was autoclaved before carrying out the experiments. The samples were characterized using dynamic light scattering (DLS) (Malvern Panalytical), UV-VIS (UV-1800, Shimadzu), SEM (Zeiss EVO 18 448), and XRF (Shimadzu ED7000) techniques. The scheme at Fig. 1 below represents the steps involved in the present work.

Synthesis of TiO_2 Nanoparticles

TiO_2 nanoparticles synthesis was carried out with a laboratory-based method in which reducing agent nitric acid and precursor TTIP was used. The mixture was prepared in a ratio of 1:1:4 using TTIP, ethanol, and DW. Further, this mixture was stirred continuously for 30 minutes at room temperature. pH of the solution was maintained at neutral and kept undisturbed for 24 hours to carry out the aging process. The resultant was then autoclaved for 2 hours at 120°C. Thereafter, the resultant was cooled at room temperature and washing was done with DW to remove impurities. The final product was obtained when the resultant was autoclaved for 2 hours at 450°C and then cooled at room temperature followed by fine grinding.[1]

Sample Preparation of Rexine, Paint, and Glass

The rexine samples were prepared from a car

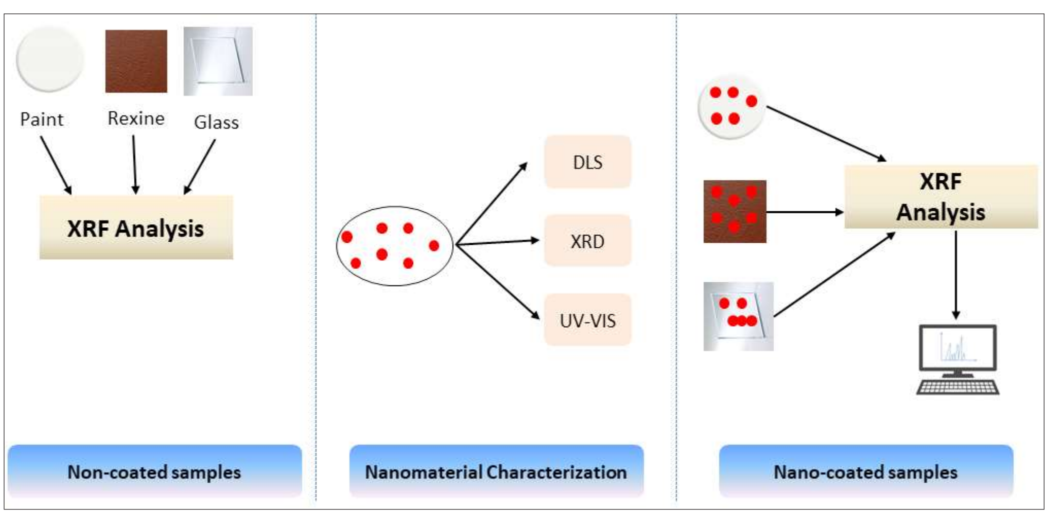

Figure 1 The Scheme represents the three techniques involved in the characterisation of samples.

cover sheet. The sample preparation of the paint was done on the surface of an aluminum sheet. The samples were divided into two parts where one part was coated using TiO_2 nanoparticles and the other was used for the control or comparison observation. Before analysis, the surface was carefully washed with DW to remove any impurities and was dried. One part of the sample was coated using TiO_2 nanoparticles.

For rexine, a solution was prepared in DW using TiO_2 nanoparticles with a concentration of 0.4 mg/mL, and the solution was then sonicated for 30 minutes. Further, it was immersed in this solution for 10 minutes. The deposition of TiO_2 nanoparticles on the surface was followed by annealing at 75°C for 60 minutes. The samples were then washed with DW in an ultrasonic bath to remove undeposited nanoparticles.

For the paint sample, the aluminum sheets were coated with paint in the presence of TiO_2 nanoparticles when the paint was dried and the flakes were then used for the analysis. Similarly, the glass samples were prepared on a glass slab while coating the TiO_2 nanoparticles on the slab surface. Figures 1(a), (b), and (c) show rexine, paint, and glass samples.

Characterization of Prepared Samples

The synthesized TiO_2 nanoparticles were characterized using different techniques. DLS was done for determining the size of the particles. To carry out the DLS analysis 1 mg/mL solution was prepared and was placed in a cuboid to determine the nanoparticle size. The XRD analysis was carried out for obtaining the crystallinity of the material (a 2mg powder sample was used for XRD analysis). The optical properties of the synthesized material were determined with UV-VIS. To perform the UV-VIS analysis a dilutes sample was prepared and was kept in the spectrophotometer. Further, the sample prepared on different surfaces like rexine, paint, and glass were characterized using SEM to study the surface of the sample and XRF analysis to determine the elemental composition of the prepared sample.

RESULTS

The size of the prepared sample was determined using DLS performed at Amity Institute of Nanotechnology, Amity University Uttar Pradesh, Noida. The size of the nanoparticles was obtained within 500 nm as shown in Figure 2(a). The XRD analysis was performed for TiO_2 nanoparticles and the recorded spectrum

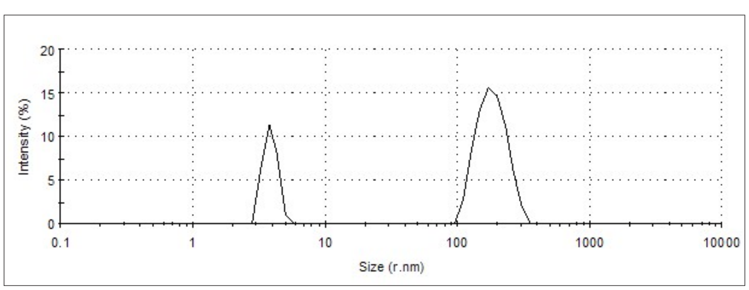

Figure 2: Samples used for analysis (a) Cotton (b) Paint (c) Glass

is illustrated in Figure 2(b). The diffraction peaks were obtained at 25.63°, 27.62°, 36.33 °, 48.39°, 54.50° confirms the synthesis of TiO2 nanomaterials[2]. Further, the UV-VIS analysis of the TiO2 nanoparticles was carried out to determine the optical properties as shown in Figure 2(c). The absorbance was obtained at room temperature and in a nano range. The sample was scanned within a wavelength ranging from 200 to 700nm. It was observed that a peak was shown at a wavelength of 312 nm showing good absorbance in the ultra visible region.[3]

Scanning Electron Microscopy

The surface of the nanoparticle-coated samples was characterized using scanning electron microscopy (EVO 18 Special Edition, Zeiss). Figure 3 (a, b) illustrates the SEM micrographs for glass and rexine samples respectively coated with TiO_2 nanoparticles. The spherical structure on the surface of the rexine, paint, and glass slab was observed in nanometer size 4-6. Hence, it was confirmed that the TiO_2 nanoparticles are present in the glass and rexine surfaces.

X-Ray Fluorescence Spectroscopy

The rexine, paint, and glass samples were examined using the XRF technique (Shimadzu ED7000). The samples were placed directly in the chamber with air atmosphere one after other. For operating the instrument a voltage of 50 KV was used and an acquisition range from 0-40 Kev. For target material rhodium (Rh) was incorporated. The sample was analyzed in the same target area in both samples.

XRF Analysis for Rexine Samples

The elemental analysis for rexine is displayed in Figure 4(a) and (b) for non-coated and coated samples respectively. The elemental profile obtained during analysis is shown in Table 1 (a) and (b) for coated and non-coated samples respectively. The obtained results show the presence of a higher percentage of chlorine (Cl) (83.83%) followed by titanium (Ti) (6.84%) and calcium (Ca) (6.67%) in both samples. The observed intensity peak exhibits no significant difference for Ti elements in both samples. This may be due to the presence of TiO_2 particles in the rexine sample having higher concentrations rather than the coated TiO_2 nanoparticles.

Determining the Composition of Paint Sample using XRF Technique

The XRF analysis carried out for the paint sample is illustrated in Figure 5. The titanium peak obtained in both the samples (non-coated and TiO_2 nanoparticle-coated) is almost similar. The XRF graph for non-coated samples is shown in Figure 5(a) whereas, the nanocoated is shown in Figure 5(b). Table 2(a) shows the elemental percentage of composition of non-coated samples and for nanocoated samples,

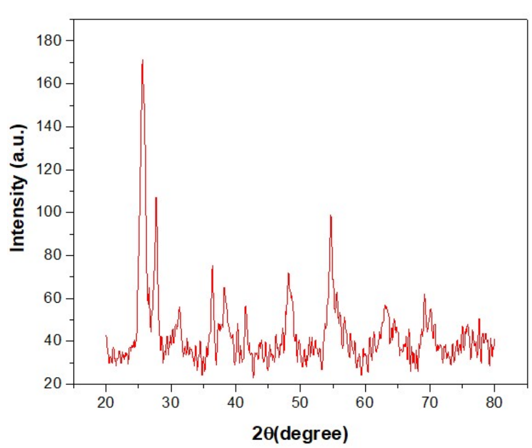

Figure 2(b)

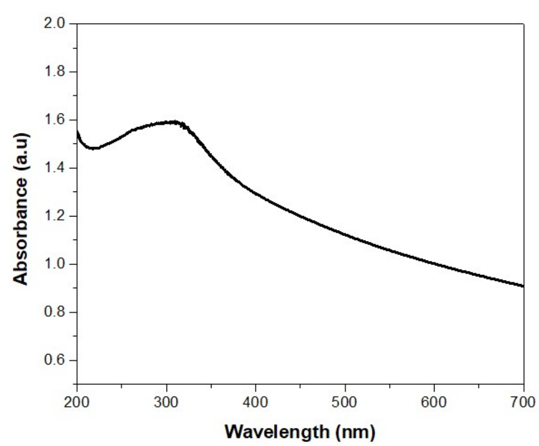

Figure 2(c)

the results are shown in Table 2(b). The sample is composed of approximately 98% for both the samples, no large difference is obtained between the titanium percentage for non and nanocoated paint samples. This was due to the presence of white color paint coated on the surface of the aluminum sheet itself containing TiO_2 particles.[7]

Analysing the Glass Sample using the XRF Technique

The surface of the glass slab was characterized under the XRF technique to study the presence of elements and their percentage on the surface. Figure 6 depicts the XRF analysis for non-coated [Figure 6(a) and nanocoated (Figure 6(b)] samples of glass slabs. A higher percentage of silicon (Si) (64.58%) was observed followed by Ca (30.7%) in both samples as shown in Table 3(a) and (b). The principal constituent of glass is silica (SiO_2) and calcium oxide (CaO) which are used to increase their durability. Whereas, the percentage of Ti was very low (0.20%) in the case of non-coated glass slab and was higher (2.02%) in the nanocoated sample. Also, a large difference was observed in peak intensity of Ti from the control sample (0.373 cps/µA) to nanoparticle coated (3.661) differentiating the two samples.

DISUCSSION

The most common piece of evidence that can be frequently collected from the crime spot for forensic analysis are fiber, fabrics, hair, paint, and glass. The evidence in cases like rape, burglary, and physical violence can be collected from the various materials at the crime scene. Presently, the evidence collected for solving the forensic analyses is mostly in the form of bloodstains and fingerprints which are then analyzed using spectroscopic techniques.[8,9] The forensic analysis of these materials is difficult due to the complex chemical composition of samples.[9]

With the new advances in technology, the textile is nowadays coated with nanomaterials. They are used for digital printing, textile coloring, and developing smart fabrics as they provide various exceptional properties such as self-cleaning, anti-bacterial, thermal retardancy, ultra-violet protection, and others.[10-15]

The imaging techniques used in surface analysis for nanomaterial characterization are Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Scanning Tunneling Microscopy (STM), and Atomic Force Microscopy (AFM).[16-18] The vibration mode of molecules is studied with spectroscopic techniques including Fourier Transform Infrared (FTIR) spectroscopy and Fourier Transform Raman (FT-RAMAN) spectroscopy.[19,20] Ultraviolet-visible spectroscopy (UV-VIS) technique is

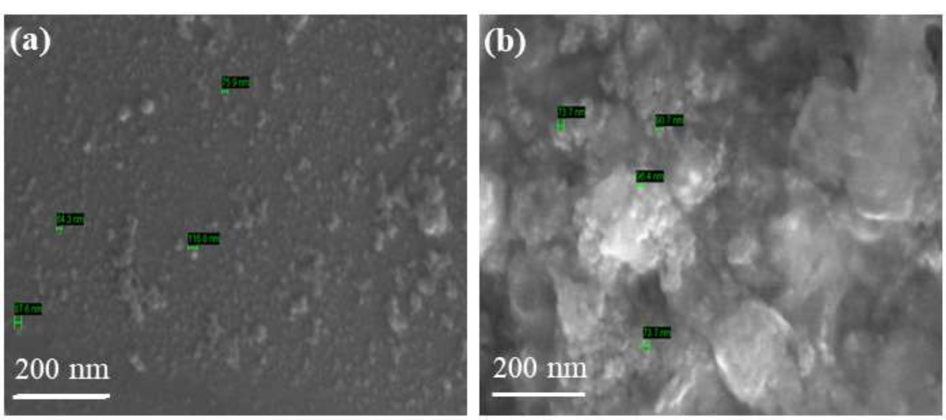

Figure 3: Scanning electron microscopy for TiO2 nanoparticle coated samples at 200 nm: (a) Cotton; (b) Glass; (c) Paint; (d) Rexine

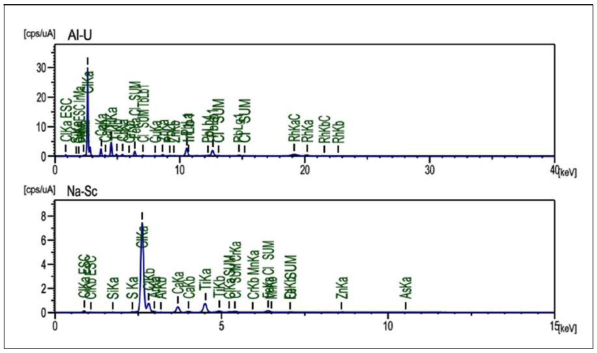

Figure 4(a):

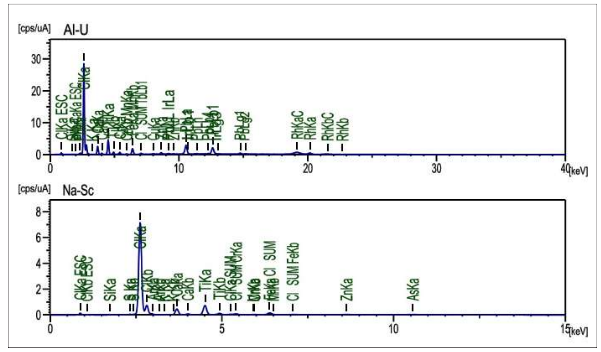

Figure 4(b):

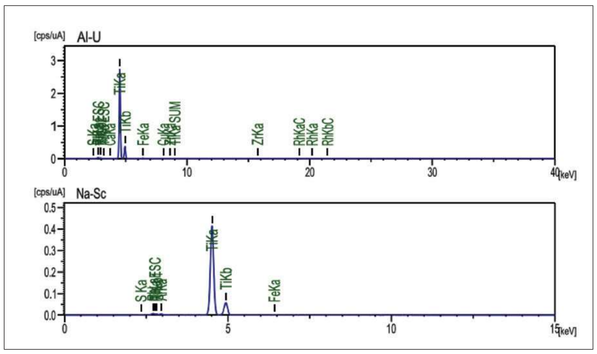

Figure 5(a):

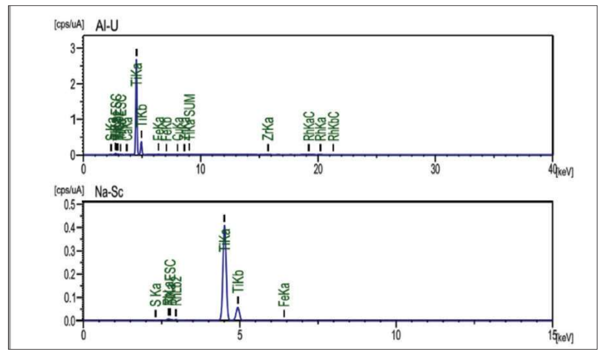

Figure 5(b):

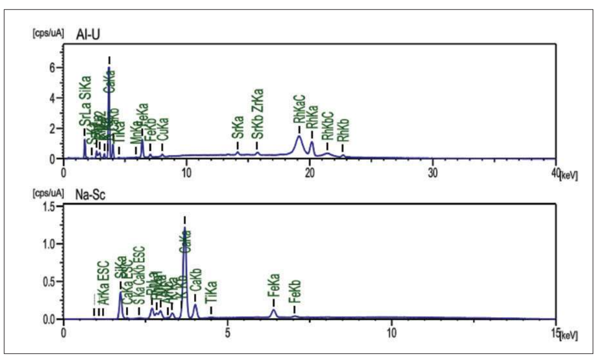

Figure 6(a):

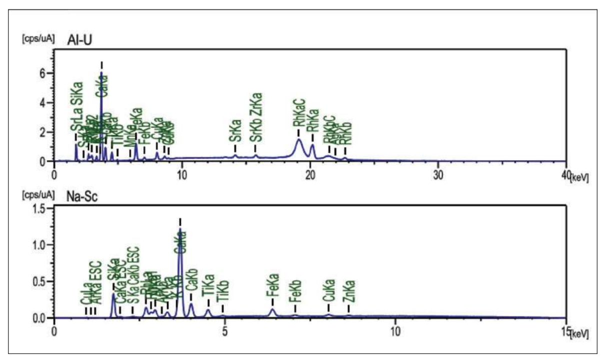

Figure 6(b):

used to study the optical properties of the nanomaterials.[21] X-ray techniques like X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) spectroscopy, and X-Ray Photoelectron Spectroscopy (XPS) are used to get information about the crystalline structure and oxidation state of the elements of nanomaterials.[22,23] Among these techniques, XRF is well known for elemental analysis in forensic investigation.[24] In an X-ray, when allowed to hit the surface of the material, electrons are ejected from the atom of an element present in the sample resulting in determining their composition. This technique facilitates in retrieving information about the elemental composition of the samples obtained from the crime scene. Samples such as paint, glass, and textile are very common in cases including murder, rape, and burglary. It can provide better analysis of these samples.[25-27] There are various advantages

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1	Cl	83.83	0.225	Quant.-FP	ClKa	75.910
2	Ti	6.84	0.059	Quant.-FP	TiKa	30.465
3	Ca	6.67	0.067	Quant.-FP	CaKa	5.139
4	Pb	0.85	0.008	Quant.-FP	PbKa	18.630
5	Si	0.59	0.058	Quant.-FP	SiKa	0.100
6	Cr	0.52	0.011	Quant.-FP	CrKa	4.311
7	Fe	0.32	0.004	Quant.-FP	FeKa	4.848
8	S	0.19	0.014	Quant.-FP	S Ka	0.255
9	Zn	0.11	0.004	Quant.-FP	ZnKa	3.683
10	Cu	0.03	0.003	Quant.-FP	CuKa	0.936

Table 1(a): X-ray Fluorescence (XRF) elemental analysis for rexine samples (a) without nanoparticle coating

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1.	Ti	98.35	0.418	Quant.-FP	TiKa	16.644
2.	Fe	0.72	0.175	Quant.-FP	FeKa	0.009
3.	S	0.57	0.029	Quant.-FP	S Ka	0.082
4.	Cu	0.092	0.023	Quant.-FP	CuKa	0.014
5.	Ca	0.091	0.019	Quant.-FP	CaKa	0.024
6.	Zn	0.091	0.005	Quant.-FP	ZnKa	0.068
7.	Zr	0.07	0.017	Quant.-FP	ZrKa	0.023

Table 2(a): XRF spectroscopy for paint samples (a) without nanoparticle coating

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1.	Si	64.58	0.643	Quant.-FP	SiKa	3.149
2.	Ca	30.71	0.144	Quant.-FP	CaKa	13.953
3.	K	2.36	0.042	Quant.-FP	K Ka	0.765
4.	Fe	1.26	0.017	Quant.-FP	FeKa	8.637
5.	S	0.61	0.061	Quant.-FP	S Ka	0.090
6.	Ti	0.20	0.024	Quant.-FP	TiKa	0.373
7.	Cu	0.11	0.007	Quant.-FP	CuKa	1.382
8.	Sr	0.05	0.003	Quant.-FP	SrKa	2.018
9.	Zr	0.046	0.003	Quant.-FP	ZrKa	1.715
10.	Mn	0.045	0.010	Quant.-FP	MnKa	0.226

Table 3(a): Elemental composition of glass samples using XRF (a) non-coated;

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1.	Cl	82.72	0.228	Quant.-FP	ClKa	72.517
2.	Ti	6.78	0.059	Quant.-FP	TiKa	29.623
3.	Ca	6.74	0.069	Quant.-FP	CaKa	5.100
4.	Pb	0.93	0.009	Quant.-FP	PbKa	19.607
5.	Fe	0.86	0.011	Quant.-FP	FeKa	12.567
6.	Si	0.82	0.066	Quant.-FP	SiKa	0.136
7.	Cr	0.52	0.011	Quant.-FP	CrKa	4.239
8.	S	0.23	0.013	Quant.-FP	S Ka	0.296
9.	K	0.12	0.025	Quant.-FP	K Ka	0.059
10.	Zn	0.11	0.004	Quant.-FP	ZnKa	3.605
11.	Cu	0.04	0.003	Quant.-FP	CuKa	1.170
12.	Ir	0.03	0.007	Quant.-FP	IrKa	0.434
13.	Mn	0.02	0.006	Quant.-FP	MnKa	0.272

Table 1 (b): X-ray Fluorescence (XRF) elemental analysis for rexine samples (b) surface coated with TiO2 nanoparticles

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1.	Ti	98.33	0.422	Quant.-FP	TiKa	16.314
2.	Fe	0.64	0.032	Quant.-FP	FeKa	0.091
3.	S	0.62	0.182	Quant.-FP	S Ka	0.008
4.	Cu	0.10	0.019	Quant.-FP	CuKa	0.027
5.	Ca	0.10	0.023	Quant.-FP	CaKa	0.016
6.	Zn	0.09	0.018	Quant.-FP	ZnKa	0.028
7.	Zr	0.08	0.004	Quant.-FP	ZrKa	0.060

Table 2(b): XRF spectroscopy for paint samples (b) with TiO_2 nanoparticle coating

S.No.	Analyte	Result (%)	Std. Dev	Calc. Proc.	Line	Intensity (cps/μA)
1.	Si	64.04	0.640	Quant.-FP	SiKa	2.925
2.	Ca	31.00	0.146	Quant.-FP	CaKa	14.052
3.	K	2.35	0.041	Quant.-FP	K Ka	0.766
4.	Ti	2.02	0.043	Quant.-FP	TiKa	3.661
5.	Fe	1.25	0.018	Quant.-FP	FeKa	8.246
6.	S	0.63	0.060	Quant.-FP	S Ka	0.094
7.	Cu	0.30	0.009	Quant.-FP	CuKa	3.688
8.	Zn	0.106	0.007	Quant.-FP	ZnKa	1.502
9.	Ag	0.104	0.013	Quant.-FP	AgKa	0.952
10.	Sr	0.06	0.004	Quant.-FP	SrKa	2.005
11.	Zr	0.05	0.004	Quant.-FP	ZrKa	1.802
12.	Mn	0.04	0.012	Quant.-FP	MnKa	0.231

Table 3(b): Elemental composition of glass samples using XRF (b) TiO2 nano-coating

associated with XRF analysis such as it is non-destructive, provides rapid results, and is easy to use. Therefore in the present work, the XRF technique is used for analyzing the elemental composition of various materials attributed to forensic investigations.

Various nanoparticles have been used in the field of forensic investigation such as gold nanoparticles which are used for storing fingerprints for a long period and possess several advantages such as high sensitivity, selectivity, and inert nature.[28-32] The silver nanoparticles are used for enabling the visualization of fingerprints during criminal investigations.[33-35] Zinc oxide nanoparticles (ZnO-NPs) have excellent properties such as high excitation binding energy, and wide-band. Therefore, ZnO-NPs are used in the form of nano-powder to obtain latent fingerprints.[36-39] Silica Nanoparticles (SiO_2-NPs) are used for interaction with an organic compound present on the surface, besides these nanoparticles prevent photo-decomposition of the fingerprints.[40] It has been reported that titanium dioxide is used in forensic applications such as latent fingerprinting or fingermark powder because they help in decreasing the risk level during inhalation and have low toxicity for investigators.[41]

CONCLUSION

In this study, the effective analysis of distinct surfaces of rexine, paint, and glass coated with nanomaterials was performed by using XRF analysis. The study was performed with two sets of observations.

Firstly, the surface of the material was analyzed without nanoparticle coating and then the surface of the material was coated with TiO_2 nanoparticles. TiO_2 are widely used in consumer products and have several applications in forensic science due to their efficacy in fingerprinting and reducing the risk level of inhalation during investigations. Hence, TiO_2 nanomaterials were synthesized and characterized by using different techniques such as DLS, XRD, and UV-VIS. Further, the synthesized TiO_2 nanoparticles were then coated on the surface of rexine, paint, and glass. Thereafter, the surface of these samples was characterized by using SEM, and the elemental composition of these samples was obtained with XRF analysis. It was observed that a notable difference was obtained in the titanium concentration with the glass sample.

In the case of other samples (rexine and paint), a minimal difference was observed indicating the presence of higher composition of titanium which was already present during their manufacturing process. The study presented an effective approach to exhibiting the integration of nanotechnology that can play a vital role in forensic investigation to provide selective and sensitive ways for detecting and solving criminal cases with infallible evidence. IJFMP

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Conflict of Interest:
The authors declare that there is no commercial or financial links that could be construed as conflict of interests.
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