Identification of a tiny, thin and smeared dot of red paint in a fatal traffic accident case by Fourier transform-infrared microspectroscopy

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ABSTRACT

Fourier transform-infrared microspectroscopy (micro-FT-IR) operated in attenuated-total-reflectance (ATR) and transmission modes following stereomicroscopy was used for the purpose of matching known and questioned paint samples in a fatal hit-and-run case. A tiny, thin and smeared dot of red paint sticking on the broken-off cross section of a side-mirror rod was taken from the victim motorcycle and referred to as the questioned sample. Two sets of red paint chips were collected as known samples from two suspect gravels trucks respectively. These were analyzed to determine whether the red paint dot found in the motorcycle could have been the transfer from one of the two gravels trucks. Although the known and questioned paint samples are in different sample forms and the grayish white polymeric background beneath the thin red paint dot somewhat interfere with the spectra comparison, the results obtained confirm the questioned red paint dot from the motorcycle and the red paint chips from one of the trucks are of the same class of paint; that is, the possibility of the paint transfer cannot be eliminated.

Keywords: forensic science, paint examination, automotive paint, hit and run, Fourier transform-infrared spectroscopy, infrared microscope, attenuated total reflection, stereomicroscopy

Introduction

Based upon Locard's principle of exchange, automotive paint chips or smears are one of the most common types of physical evidence received by forensic science laboratories for analysis and in most traffic accident cases have eventually shown considerable evidential values. However, the complexity of modern paint chemistry and automotive finishes, together with the chemical inertness of many paints, tend to frustrate the forensic scientist, resulting in problems in deriving maximum information from paint evidence [1-2]. Moreover, forensic paint samples are often analyzed in limited amounts that preclude the application of standard paint analysis procedures or protocols. Depending on the issues under investigation, sequence of events at the scene, complexity and condition of the samples, envi-ronmental effects, and collection methods, a criminalist has often been forced to address such issues as test choice, sample preparation scheme, test sequence, and degree of sample alteration and consumption, which are efficacious to each specific case as well as the interests of all parties, to a litigation [3].

Both known and questioned paint specimens should be properly collected for a forensic paint analysis. Ideally, whole paint flakes provide the most useful questioned samples. Although fragments of films that may or may not represent the complete lateral cross-sectional structure of the original finish offer fewer characteristics for comparison, they can be useful in physical fits and other examination [3]. If the topcoat can be defined, then a conclusive comparison to known specimens may be possible. In contrast, smeared or abrasively transferred samples may also provide useful information, but

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they are generally far more difficult to analyze. Smeared transfers can exhibit the pigments and the vehicles mingling from different paint layers or films that could preclude application of those standard analytical methods described in the forensic science textbook or laboratory manual. We have previously reported the identification of tiny, thin and scattered smears of automotive paint in a fatal and controversial traffic accident case using three complementary methods in the order of stereomicroscopy, micro-FT-IR spectroscopy and solubility tests [4]. This paper deals with a fatal hit-and-run case involving a tiny and thin dot of red paint.

Background

On the afternoon of 20 May 2000 in Taoyuan (Taiwan, ROC) an autobike rider was killed when he, along with his autobike, collided with an overpassing gravels truck. Following the witnesses' descriptions, two suspect gravels trucks A and B were caught and appropriate red paint chips or films collected from relevant facial areas as ten sets of known samples (designated as "A-1" through "A-8" and "B-1" and "B-2", respectively). These together with a tiny and thin dot of red paint sticking on the broken-off cross section of a side-mirror rod taken from the victim motorcycle and referred to as the questioned sample (designated as "Q") were submitted for our examination to determine whether the red paint dot found in the motorcycle could have been the transfer from one of the two gravels trucks.

Methods of Identification

Stereomicroscopic examination

The side-by-side microscopic examination and comparison of physical features of all samples was carried out using a Zeiss Stemi SV 11 stereomicroscope with a total magnification of 66. For examining the lateral cross-sections of samples A's and B's, both the naked cross-sections and the microtomed thin-edge sections "borrowed" from those for the following transmission FT-IR tests were employed.

Micro-Fourier transform infrared spectroscopy

The micro-FT-IR analyses of the microtomed thin-edge sections (ca. 5-£gm thickness) of samples A's was performed using a Janssen Micro-FT-IR Spectrometer

operated in the transmission mode with scanning areas of 300 ¡Ñ300, 300 ¡Ñ120, and 240 ¡Ñ60 £gm2 for scanning the whole thickness of the thicker red layer, the outer and inner margins of the red layer, and the whole thickness of the thinner black layer, respectively, at a magnification of 160. Each scanned region was subjected to a single analysis. Meanwhile, the upper surfaces of samples A's, B's and Q were also directly profiled using a Nicolet Micro-FT-IR Spectrometer operated in the ATR mode with a scanning area of 30 x 30£g m2 at a magnification of 150. Samples A's were each analyzed once; samples B's were each analyzed three times; sample Q, five times toward itself and one time for each of three different spots in the appropriate vicinity of sample Q as its blank control (designated as "Q-blk"; so doing was to properly take into account the uneven thickness and possibly inhomogeneous microstructure of the questioned paint dot as well as the possibly interfering background). For any set of replicate analyses, the most representative IR spectra (i.e., those showing relatively more intense and better resolved key absorption bands) were chosen for the relevant comparisons. To make the comparison between different sample forms simple yet reasonable, we set ¡Ó10 cm-1 as the window for establishing a match of two absorption bands under comparison.

Results and Discussion

Strategy of the identification

Directed toward the above stated objective of the examination, our strategy was to search for as many as significant differences between samples A's and B's as well as between sample Q and its white background, while verifying the absence of significant differences and the presence of as many as matching features between sample Q and either samples A's or B's. The likelihood for sample Q and either samples A's or B's to be of common origin, i.e., one of the two trucks, is a function of a number of factors including the type or number, or both, of matching features, the type of components in the paint film, the presence or absence of studies quantifying the uniqueness of these components, and the discriminating power of the methods used [3]. Unfortunately, for the following three reasons we had to preclude the use of such methods as micro-spectophotometry (or surface colorimetry) [5], pyrolysis-gas chromatography (Py-GC) [6], pyrolysis-gas chro-

matography-mass spectrometry (Py-GC-MS) [7], high performance thin-layer chromatography (HPTLC) [8], scanning electron microscope/energy dispersive X-ray analysis (SEM/EDX) [3,4,9,10] and even solubility test [4,8,11], which had long been established as useful in our laboratory: (1) the extremely limited size and thickness along with the uneven thickness and possibly inhomogeneous microstructure of sample Q; (2) the unfeasibility of effectively separating sample Q from the possibly interfering background; and (3) the inherent unequal bases on which the comparative analysis would be made (i.e., samples A's were two- or three-layered paint chips showing their original finishes and samples B's were non-layering paint films whereas sample Q looked like a paint smear showing no layering structure of its original finish). Consequently, our actual analysis was started with the simplest nondestructive visual and microscopic examination followed by nondestructive infrared spectroscopy [12-15].

Macroscopic (visual) and stereomicroscopic examinations

Except for the various sizes and shapes of the paint chips that mean little to the present identification and will not be addressed, samples A-1 through A-8 have virtually the same appearance and sample B-1 and B-2 also have mutually indistinguishable external features. Table 1 summarizes other characteristics observed for the upper (outer) surfaces and/or lateral cross-sections of samples A's, B's and Q with and without magnification. Based on these observations, sample Q can be concluded to be exogenous, but not endogenous, and it's really a "red paint" dot, but not any other material. However, since the microscopic red color visions and micro-textures of samples A's, B's and Q are essentially indistinguishable from one another and paint is usually considered as "class evidence" in the absence of any "physical match" between the evidence and exemplar samples [1], it is therefore fair to say, at this point, that the possibility of sample Q being transferred from one of the two suspect gravels trucks can not be eliminated.

Micro-Fourier transform infrared spectroscopy

As was for our previously reported case concerning an identification of tiny, thin and scattered smears of automotive paint [4], although the disagreement between the original absorption data obtained via the transmission mode of Janssen Micro-FT-IR Spectrometer and the reflectance mode of Nicolet Micro-FT-IR Spectrometer has been properly corrected and correlated, the application of IR methodology to the present case inherently suffers from a logical defect. That is, the comparative analysis was based upon two unequal bases ¢wsamples A's and B's, whether two- to three-layered paint chips or non-layering paint films, were both in the form of original finish whereas sample Q was virtually a paint smear showing no layering structure of its original finish. And it is well known that any variations in the inter- or intramolecular interactions of the analyte and the matrix could result in significant wavenumber shifts and intensity changes of IR bands, making the otherwise straightforward spectra comparison complicated. What is worse is that sample Q has uneven thickness and possibly inhomogeneous microstructure and would likely suffer irregular interferences from the background Q-blk. Fortunately, after taking the measures described in last section, most of the IR data have been normalized.

Based on the foregoing criteria, no significant differences appear among the eight ATR FT-IR spectra obtained for the upper surfaces of samples A's. A sample spectrum denoted "A-ATR" is shown in Fig. 1(a). Likewise, the six ATR FT-IR spectra obtained for samples B's are considered to be all identical. A sample spectrum denoted "B-ATR" is shown in Fig. 1(b). As to the five ATR FT-IR spectra obtained for sample Q, two of them are identical; the other three are virtually identical including all well matched wavenumbers except that any one of the three contains a band with slightly different intensity from that of another. The most representative spectrum denoted Q-ATR is shown in Fig. 1(c). Here, it should be pointed out that for a reflection plate made of zinc selenide, the depth that the IR-rays of various wavelength may penetrate into such sample as paint and its like is estimated to be several submicrons to four microns. Since sample Q did show uneven paint film thickness (looked like zero to several submicrons) under the microscope, we were at first concerned about whether the five fast and arbitrary ATR FT-IR scans on sample Q's area would involve unequal IR-ray penetration distances, and hence generate too different spectra. At some places of the reflection plate-sample interface where the paint film was thicker, a beam of radiation might penetrate only partial depth of the paint film before reflection occurred. At other places of the interface where the paint film was thinner or even zero-thickness, however, a beam of radiation might strode

Table 1   Key observations of the appearance of samples A's, B's and Q with and without magnification.
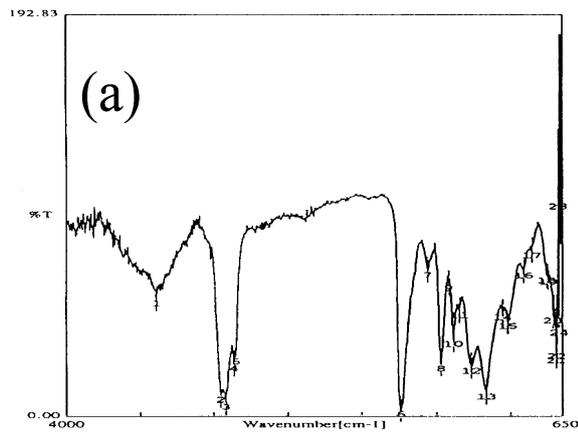
Sample	Key observations	
	Macroscopically	Under stereo microscope (66×)
Samples A-1 through A-8 (Known red paint chips taken from truck A)	1. All the sample A chips' upper (outer) surfaces are bright red. Their color visions are indistinguishable from one another. 2. Inner surfaces' colors: Samples A-1 and A-3, uneven yellowish white; sample A-2, uneven light brownish gray; sample A-4, uneven light brownish white; sample A-5, light and dark brown mixed; samples A-6 and A-8, grayish white; sample A-7, yellowish white and gray mixed. 3. It is hard to recognize visually the layering structure of the lateral cross-section. However, since the main color of the lateral surface (black) is so different from those of the outer and inner surfaces, each of the sample A chips should have at least three layers.	1. Upper view: Each of the sample A chips shows only a single bright red color without any gloss. (unlike most of the automotive finishes whose outermost paint films under the microscope show a great number of glittering micro-granules in various colors even though the macroscopic color is uniform) 2. Lateral view: Each chip shows only a thinner outer layer in bright red and a thicker middle layer in black; no clearcoat is observed; the innermost layer does not show appreciable thickness.
Samples B-1 and B-2 (Known red paint films taken from the front bumper of truck B)	1. All the sample B films look somewhat withered, and their red color looks a little faded. 2. Only one single layer can be visually recognized on the lateral cross-section.	1. Upper view: Each of the sample B films shows only a single bright red color without any gloss. [unlike most of the automotive finishes ….(the same as the above stated)] The color vision and micro-texture is indistinguishable from those of samples A's. 2. Lateral view: All the sample B films look somewhat like house paint flakes showing no layering structure. (unlike most of the automotive whole paint chips whose lateral cross-sections under the stereo microscope (66×) or micro-FT-IR (160 ×) show three to five layers, with the outermost layer being a noncolor clearcoat or a colored basecoat; the latter is usually applied as the 2nd outermost layer followed by primer-surfacer and primer layers)
Sample Q (Questioned red paint dot sticking on the broken-off motorcycle side-mirror rod)	1. Looks bright red. The color vision of the red dot is indistinguishable from that of samples A's. 2. The grayish white background of the red dot which makes the interior of the motorcycle side-mirror rod is an entirely different material, whether organic or inorganic, from the red dot itself.	1. Upper view: Appears to be a rather thin paint film showing only a single yet uneven bright red color without any gloss. [unlike most of the automotive finishes ….(the same as the above stated)] The color vision and micro-texture is indistinguishable from those of samples A's and B's. 2. Lateral view: Shows no layering structure of the original finish.

across the total paint film thickness plus partial depth of the background material or might penetrate directly into partial depth of the background material. It turns out no significant differences appear among the five ATR FT-IR spectra for sample Q, probably benefiting from the averaging effect of a great number of scans and total reflection processes within a specific scanned area. Also, the three ATR FT-IR spectra obtained by scanning sample Q's three nearby spots as a measurement of Q-ATR's background were recognized as all identical [Q-blk-ATR; Fig. 1(d)]. Thus, generally speaking, the ATR-mode FT-IR employed in the present case provides high reproducibility and hence strong discriminating power. However, probably because our IR library of automotive paint comprises transmission-mode spectra for basecoats only, none of the above obtained three evidence spectra, A-ATR, B-ATR and Q-ATR, matches any pre-existing standard spectra in our IR library, nor do the three spectra, after going through the "automotive paint binder infrared classification flow chart" described by Bartick et al. [9], find any matched objects of automotive paint binder.

Also based on the foregoing criteria for spectra comparison while keeping in mind the possible interference from Q-blk-ATR with Q-ATR, we conclude that Q-ATR is totally different from B-ATR and completely eliminate the possibility of the red paint dot found in the motorcycle being the transfer from gravels truck B. Among all the IR spectral differences, the most evidential is that Q-ATR shows a very strong and characteristic carbonyl stretching band at ca. 1717 cm-1 whereas B-ATR does not. In contrast, the spectral pattern of Q-ATR looks very much like that of A-ATR, although there are a few minor differences. A closer comparison of these two spectra indicates that most of the characteristic absorption bands in A-ATR (obtained from original paint, thereby serving as the subject of comparison) such as 3500~3100 (m), 2930 (m), 2864 (w), 1723 (s), 1537 (m), 1490 (w), 1385 (w), 1138 (s), 1071 (s), 848 (m), 762 (s), and 700 cm-1 (s) (where "s" stands for "strong"; m, moderate; w, weak) are also found in Q-ATR. Although such also characteristic bands of A-ATR as 1685 (s), 1461 (s), 1238 (m), 1019 (m), and 995 cm-1 (m) are not duplicated in Q-ATR with exactly the same wavenumbers and intensities, they do appear in Q-ATR with only minor variations in wavenumber, intensity and/or shape. These minor variations are mostly considered as reasonable and within acceptable ranges of accuracy if we recall the inherent differences between samples A and Q

in sample form and micro-structure as well as the possible spectral interference from Q-blk with Q. The only bands in A-ATR that may be controversial are 1723 (s) and 1685 cm-1 (s). These two neighboring bands' counterparts in Q-ATR become so overlapped that the instrument can label a single wavenumber only, i.e., 1717 cm-1 (s). This question shall be clarified later with the aid of transmission-mode FT-IR. Finally, referring also to Q-blk-ATR, we attribute the weak yet broad band 2700~2200 cm-1 (w), which appears in Q-ATR only, as well as other minor spectral variations between Q-ATR and A-ATR to the interference with sample Q from its background.

To further investigate whether the two neighboring bands in A-ATR, 1723 (s) and 1685 cm-1 (s), become largely overlapped at ca. 1717 cm-1 in Q-ATR is reasonable or not, we also performed the transmission-mode FT-IR on the microtomed thin-edge sections of the eight samples A's according to the procedure described in last section. The eight spectra obtained for the eight red layers' inner margins are all identical, with a sample spectrum denoted "A-Tra/red-in" and shown in Fig. 2 (a); the eight spectra obtained for the eight red layers' outer margins are all identical, with a sample spectrum denoted "A-Tra/red-out" and shown in Fig. 2(b); the eight spectra obtained for the eight red layers' whole thicknesses are all identical, with a sample spectrum denoted "A-Tra/red-whole" and shown in Fig. 2(c); the eight spectra obtained for the eight black layers' whole thicknesses are all identical, with a sample spectrum denoted "A-Tra/black-whole" and shown in Fig. 2(d). After cross-comparing these lateral-view transition-mode spectra for samples A's layers or sub-layers' and the foregoing upper-view ATR-mode spectrum for samples A's [i.e., A-ATR; Fig. 1(a)], we can find: a) As has been expected, spectra A-Tra/red-out and A-ATR are well matched in most of their spectral features. In particular, they both show relatively better resolved doublet-bands around 1700 cm-1. They also demonstrate that the small difference in their doublet-bands' relative intensi1es does not necessarily mean they have resulted from different classes of paint, since IR spectra's wide bandwidths have made their band intensities multi-factors dependent; b) Spectra A-Tra/red-in, A-Tra/red-out, and A-Tra/red-whole are extremely similar to one another in most of their spectral features, but, in terms of the resolution of the doublet-bands around 1700 cm-1, they are not the same; namely, the doublet-bands around 1700 cm-1 is the best resolved in A-Tra/red-out, the most overlapped in A-Tra/red-in, and about the average in A-

Fig. 1 The ATR-mode micro-FT-IR spectra obtained upon scanning the upper surfaces of samples A's (a), B's (b), Q (c), and the background of sample Q, i.e., Q-blk (d).

(a)

Parameter
Accumulation   512 times
Resolution   4 cm-1
Apodization   Cosine
Date/Time   2001/6/13 3:20PM
Sample Name
WHITE
Gain   100
Aperture   mm
Zero Filling   x2
Speed   10.0 mm/sec
Delay Time   0   sec
File Name   NONAME80.J1D
Light Source   Standard
Detector
Beam Splitter   KBr

Peak Information

1	3396.03	56.2%	2	2960.20	10.0%
3	2926.45	6.6%	4	2871.49	24.9%
5	2857.02	28.4%	6	1734.66	3.0%
7	1551.45	70.1%	8	1462.74	24.8%
9	1409.71	63.5%	10	1378.85	36.6%
11	1337.39	50.5%	12	1255.43	23.8%
13	1159.01	11.4%	14	1048.12	49.5%
15	1011.48	45.2%	16	904.45	69.8%
17	850.45	79.7%	18	748.25	67.4%
19	736.67	66.6%	20	701.00	47.8%
21	678.82	28.9%	22	674.96	30.8%
23	668.21	103.3%	24	659.54	41.9%

(b)
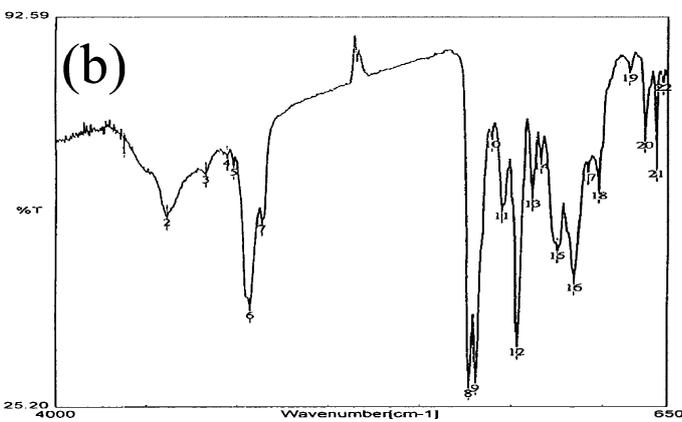
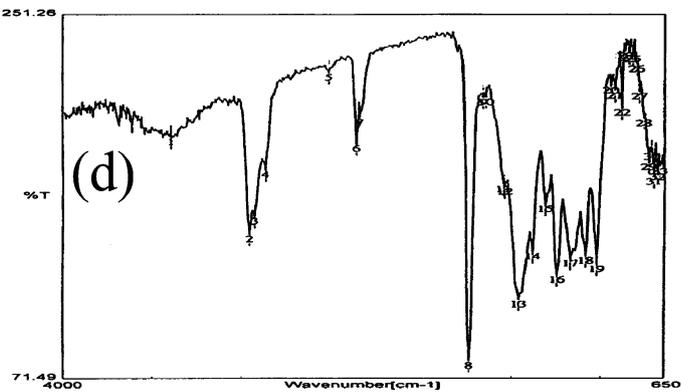Parameter
Accumulation   221 times
Resolution   4 cm-1
Apodization   Cosine
Date/Time   2001/8/2 3:13PM
Sample Name
blue
Gain   20
Aperture   mm
Zero Filling   x2
Speed   10.0 mm/sec
Delay Time   0 sec
File Name   1602REDP.J1D
Light Source   Standard
Detector
Beam Splitter   KBr

Peak Information

1	3630.34	70.5%	2	3393.14	57.9%
3	3179.08	65.4%	4	3063.37	68.3%
5	3025.76	66.7%	6	2933.20	41.8%
7	2871.49	56.9%	8	1726.94	28.3%
9	1690.30	29.1%	10	1602.56	71.5%
11	1550.49	59.1%	12	1464.67	35.4%
13	1382.71	61.2%	14	1334.50	67.7%
15	1244.83	52.2%	16	1150.33	46.6%
17	1075.12	65.8%	18	1016.30	62.6%
19	848.53	83.0%	20	763.67	71.4%
21	701.96	66.4%	22	663.39	81.4%

(c)
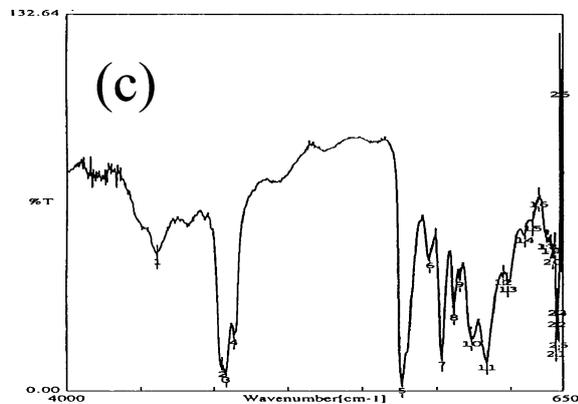
Parameter
Accumulation   512 times
Resolution   4 cm-1
Apodization   Cosine
Date/Time   2001/6/13 11:51AM
Sample Name
WHITE
Gain   50
Aperture   mm
Zero Filling   x2
Speed   10.0 mm/sec
Delay Time   0   sec
File Name   NONAME75.J1D
Light Source   Standard
Detector
Beam Splitter   KBr

Peak Information

1	3392.17	46.9%	2	2953.45	7.2%
3	2924.52	5.3%	4	2871.49	18.7%
5	1728.87	1.5%	6	1541.81	45.6%
7	1458.89	10.8%	8	1377.89	27.2%
9	1338.36	38.8%	10	1259.29	18.1%
11	1159.01	9.6%	12	1046.19	39.7%
13	1013.41	37.1%	14	895.77	54.6%
15	844.67	58.8%	16	804.17	67.4%
17	750.17	52.4%	18	736.67	52.3%
19	721.25	50.7%	20	704.85	46.9%
21	680.75	14.4%	22	674.96	24.9%
23	672.07	28.9%	24	667.25	28.7%
25	663.39	17.6%	26	655.68	106.5%

(d)

Parameter
Accumulation   219 times
Resolution   4 cm-1
Apodization   Cosine
Date/Time   2001/8/3 3:34PM
Sample Name
blue
Gain   20
Aperture   mm
Zero Filling   x2
Speed   10.0 mm/sec
Delay Time   0 sec
File Name   1903B03P.J1D
Light Source   Standard
Detector
Beam Splitter   KBr

Peak Information

1	3397.96	190.9%	2	2959.23	142.7%
3	2932.23	151.5%	4	2871.49	174.7%
5	2515.69	222.8%	6	2361.41	187.5%
7	2341.16	200.5%	8	1732.73	79.7%
9	1654.62	210.5%	10	1638.23	210.6%
11	1542.77	167.4%	12	1534.10	166.2%
13	1455.03	110.1%	14	1376.93	133.9%
15	1305.57	157.5%	16	1239.04	122.3%
17	1163.83	130.5%	18	1077.05	131.8%
19	1016.30	127.6%	20	939.16	216.4%
21	915.06	213.9%	22	877.45	205.4%
23	858.17	233.6%	24	837.92	232.6%
25	816.71	231.9%	26	797.42	227.0%
27	781.03	213.5%	28	756.92	200.2%
29	728.96	178.5%	30	713.53	183.5%

Fig. 2 The transmission-mode micro-FT-IR spectra obtained for samples A's by scanning their lateral red layers' inner margins (a), red layers' outer margins (b), red layers' whole thicknesses (c), and black layers' whole thicknesses(d).

Tra/red-whole; c) In terms of the resolution of the doublet-bands around 1700 cm^{-1}, A-Tra/black-whole has a single strong peak over there and is more similar to A-Tra/red-in than to A-Tra/red-whole, but is different from the doublet-bands containing A-Tra/red-out; d) Just like A-Tra/red-whole is a hybrid of A-Tra/red-out and A-Tra/red-in, Q-ATR [Fig. 1(c)] is also a hybrid of A-Tra/red-out, A-Tra/red-in, and even A-Tra/red-whole, strongly suggesting sample Q, due to the collision, be sort of smeared paint exhibiting binders mingling from the different red sub-layers and even the black layer in an inward direction; e) Judging from the above shown degrees in which the relevant transmission-mode spectral features for the three layers or sub-layers of samples A's vary plus the possible ATR-mode spectral interference from Q-blk with Q, it is safe to say that all the spectral variations among the evidence spectra Q-ATR, A-ATR, and A-Tra/red's, including the different resolutions of the doublet-bands around 1700 cm^{-1}, are within the "within-sample" variation of samples A's; in other words, sample Q and samples A's are of the same class of paint.

Conclusions

Micro-FT-IR operated in the ATR and transmission modes following stereomicroscopy has proved very useful in matching a tiny, thin and smeared dot of red paint in question with known paint chips. Although the known and questioned paint samples are in different sample forms and the grayish white polymeric background beneath the red thin paint dot somewhat interfere with the IR spectra comparison, the above presented results confirm: a) The thought-to-be paint dot sticking on the victim motorcycle's side-mirror rod is really exogenous smeared paint, but not any other kind of material; b) The questioned red paint dot from the motorcycle and the red paint chips from gravels truck A are of the same class of paint. In other words, the possibility that the red paint dot on the victim motorcycle has been transferred from gravels truck A cannot be eliminated.

Acknowledgments

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