SUBSTANCES WHICH AFFECT PHOTOGRAPHIC PLATES IN THE DARK

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The effect which some substances have on photographic plates in the dark has been studied with the idea of devising a method for identifying the ingredients in certain mixtures often presented for microchemical analysis.

HISTORICAL REVIEW

In 1842 Moser (1) noticed that when certain bodies were brought in contact with a silver iodide plate in the dark the developed plate showed an image, the silver iodide being darkened most where the action of the body was strongest. He concluded that all bodies may possibly be considered "self-luminous."

In 1857 and 1859 Niepce de Saint Victor (2, 3) recognized the effect produced on silver bromide paper by certain substances, such as paper, marble, chalk, feathers, and cotton, when they had been exposed to light before their contact with the sensitive paper. He found that this action would pass through a layer of air 1 cm. thick and could also penetrate collodion and gelatin but was stopped by glass, minerals, and certain varnishes. He believed that the light "was absorbed and conserved" by these bodies to be sent out later in the form of radiations.

Apparently the subject was not studied further for thirty-eight years. In 1896 Colson (4, 5) found that zinc, cadmium, and magnesium affected the photographic plate in the dark. He described this phenomenon to the emanation of metallic vapors. Tin, iron, copper, and lead were found to be inactive.

Pellat (6) in 1896 and Thomson (10) in 1897 agreed with Colson that vapors given off from the metallic surfaces were responsible for the effects obtained. In order to confirm this assumption, Thomson passed an air-blast between the sensitive surface and the metal. This produced a distortion of the image.

In 1896 MacKay (7) observed the darkening of a photographic plate when it was brought into a strong electrical field with an iron object on its surface. He ascribed this effect to magnetism. At the same time Packer (9) found that a metal became active toward the photographic film on exposure to light only when it had not previously come in contact with a conductor of electricity.

Arnold's account (13) published in 1897, showed that the sulfides of zinc, calcium, and barium, as well as uranium and calcium tungstates and retene exhibited photographic activity in the dark. He attributed the action of retene to Becquerel rays, but observed that the closely related substances, anthracene, anthraquinone, and phenanthrene, did not have this property. He apparently considered the activity in general to be due to some sort of radiation.

Russell began his classic work on this subject in 1897 (14). He observed that when a zinc screen was placed between a uranium compound and the photographically sensitive surface, the silver was acted upon not only by the radiations from the uranium but also by the zinc screen. This led him to make direct tests with zinc, which showed that polished zinc was more active than zinc which had been exposed to the air for some time. A glass plate interposed between the zinc and the sensitive surface stopped this action, but many substances, such as celluloid, sheet gelatin, collodion, vegetable parchment, gold beater's skin, and tracing paper, permitted its transmission. Aqueous vapor was found not to be an active agent in producing these effects. The action took place in an atmosphere of hydrogen as it did in air. Under ordinary conditions results were obtained in an atmosphere of carbon dioxide, although Russell believed that this effect was more probably due to the action of the carbon dioxide on the zinc plate. Alteration of the temperature appeared to produce marked differences in the intensity of the picture obtained, an increase in temperature greatly augmenting the activity of the zinc. A piece of mahogany 3.5 mm. thick, which had been in this form for at least 35 years, gave, after a week's exposure, a good picture. Coal, coke, sulphur, and sugar, however, exerted no action.

In 1898, Russell (15) published the results of additional experiments on a large number of substances, both organic and inorganic, conducted principally with a view to determining the active agent concerned in the darkening of the photographic plate in the absence of light. As a result of these tests he concluded "that certain metals have the property of giving off, even at ordinary temperatures, vapor which affects a sensitive photographic plate, that this vapor can be carried along by a current of air, and that it has the power of passing through thin sheets of such bodies as gelatin, celluloid, collodion. . . . ."

Lengyel (16), Tucker (17), Bothamley (18), Sperber (19), and Vincent (20) reported the results of their work in 1898 and 1899. Lengyel's conception was that hydrogen, formed in the presence of moisture on the metallic surface, might be considered the active agent. If the conditions for the formation of hydrogen were not present, the activity was absent. He considered it reasonable to conclude that the activity was produced either by a vapor emitted by the metal or by some sort of radiation. Like Colson, Tucker had observed the action of printer's ink on the plate. Sperber repeated some of Russell's experiments for the purpose of determining whether the activity was due to a vapor generated by the substance or to active rays. He concluded that the active substance produced vibrations similar to the vibrations of ultra-violet rays and, like them, chemically active. Bothamley observed the breaking-down of the latent image through the long action of turpentine and similar substances, which formed hydrogen peroxide when they oxidized in the air.

Russell (21), in 1899, confirmed his previous findings, and showed that the action of various bodies on a photographic plate in the dark was probably due to the presence of hydrogen peroxide, which appeared to be formed when the metal sheets were oxidized in moist air. On the basis of this assumption, Russell inferred that on supplying to the metallic surface more moisture than it would obtain from the atmosphere and the photographic plate, the activity would be increased. This was found to be the case. He made many tests on essential oils, which proved to be active, also the ordinary vegetable oils, such as linseed, colza, and olive. The last two oils were not as active as linseed oil. The mineral oils, on the contrary, were inactive, as were also such compounds as benzene, phenol, naphthalene, acetaldehyde, methyl alcohol, and "coal naphtha." Vincent (20) practically substantiated the observations of Russell in 1899 and concluded that the active substance must be hydrogen peroxide.

Levison (22), in 1900, found that Canada balsam, when baked hard and placed on the sensitive side of the photographic plate or separated from it by a layer of black paper, affected the plate very much as light would. In his opinion this effect could be attributed to true Becquerel rays, as it passed through the black paper, which is impervious to ordinary light.

About two years later Vignon (23) obtained pictures of coins which he had rubbed with zinc dust and exposed to the photographic plate. This effect, of course, was due to the zinc, which Russell had found to be active.

According to Villard (24), certain inorganic substances acquired under the influence of ozone an ability to act in this manner which persisted for more than a day. Bismuth and aluminium after treatment with ozone gave images, but the photographic impression was not uniform. It consisted of a series of black points on a gray background.

Graetz's tests (25, 31), reported in 1902 and 1904, demonstrated that the action of hydrogen peroxide on the photographic plate would pass through gold leaf and aluminium foil. Like Thomson, Graetz had observed that when a current of air was blown through the hydrogen peroxide vapor the photographic effect still persisted and was registered, as shown on developing, in the form of intense black points on the plate. He assumed that particles of an unknown nature must have bombarded the plate.

Bogojawlensky (28), in 1903, obtained positive results on several metals. He concluded that Russell's explanation of the phenomenon was plausible.

Schweidler (29), the same year, attempted to prove that retene possessed ionizing action, but failed to do so.

In 1903, Dony-Henault (30) confirmed Russell's tests with respect to hydrogen peroxide and satisfied himself that bodies indifferent to the photographic plate became active after being exposed to the action of hydrogen peroxide.

Blaas and Czermak (32), in 1904, again confirmed the findings of Russell. They observed that paper, wood, shellac, leather, silk, and other substances affected the plate after they had been exposed to light, and proposed designating such substances which became active after insolation as "photechisch" (light retaining) and the property itself as "photechie." These photoactive bodies, they concluded, sent out a diffuse radiation which belonged to the blue end of the spectrum.

Kufferath and Merckens (33), in 1904, and Merckens alone, in 1905 (40), obtained photographic images with magnesium, aluminium, zinc, cadmium, nickel, cobalt, and lead. No image was obtained with tin. In a vacuum, magnesium produced no effect, even when in contact with the plate. These investigators explained the darkening of the plate by the metals on the basis that the oxidation of the active metal produced hydrogen peroxide, which acted on the plate. They considered this to be due to the fact that upon its decomposition active oxygen was formed, which, in a nascent condition, changed the silver bromide of the gelatin layer to an easily reducible oxide.

Van Aubel (34), in 1904, found that colophony was active through black paper but not through a sheet of copper. Abietic acid, copal resin, and mastic resin gave similar results but in a different degree. Heating the colophony appeared to drive off the active material.

According to Schaum and Braun (35), the action of ozone on the photographic plate could be explained as a purely chemical reaction, with the formation of a silver peroxide, oxybromides, etc., or an ionic action, with the discharge of silver ions. The action of hydrogen peroxide might be explained in much the same manner.

Stöckert's test (36), in 1904, as well as those of Precht and Otsuki (38), in 1905, practically confirmed the findings of Graetz. Precht and Otsuki observed that the reaction between hydrogen peroxide and silver bromide gelatin depended largely on the temperature and that the photographic activity increased with the concentration to a certain maximum, after which it decreased.

In 1905 Campbell (41) reported the results of experiments on the influence producing "spontaneous ionization" in a closed vessel containing gas. He stated that the walls of the vessel were responsible for an ionization effect which he had been able to measure. He concluded that any such ionization in a closed vessel containing gas might be attributed to a radiation proceeding from the walls, that certain substances, such as tin, zinc, graphite, and platinum, might produce part of these radiations, analogous to the secondary radiations excited by Röntgen and other rays. Campbell suggested that all elements are radioactive to some degree and that some might be expected to give off an emanation. All efforts directly to detect such an emanation from lead, zinc, and aluminium, have failed, however.

The experiments of Kahlbaum and Steffens (42), in 1905, appear to indicate that gravity has some influence on the photographic activity of many substances. Their work showed that the action produced from the lower side of a metallic plate, as recorded by the sensitive layer after developing, was more pronounced than that produced by the upper side.

Streintz and Strohschneider (43) confirming the results obtained by earlier workers, reported in 1905 that they had found magnesium, aluminium, zinc, and cadmium to possess the property, after polishing, of producing an image on paper impregnated with potassium iodide. They believed that the decomposition of the potassium iodide was due to "the electrolytic solution-pressure in the neighborhood of the surrounding ions." Since they could detect the effect, even if the metal and paper were separated by a thin layer of air, they concluded that the ionization passed through the air. The effect obtained, they stated, had the character of a radiation and should be designated "metallic radiation."

Melander (44) was of the opinion (1905) that at ordinary temperatures all metals send out violet and ultra-violet rays, but that the rays are so weak that they can not be detected with the eye. Nevertheless, the ionizing action of these rays plays an important part in the phenomenon recorded on the photographic plate, when they strike its sensitive surface.

The work reported by Baborevsky and Vojtech (46), in 1906, demonstrated that ammonium amalgam did not emit any photographically active rays.

Campbell's (45) new tests in 1906 proved to his satisfaction that the emission of ionizing radiations was an inherent property of all the metals investigated (lead, copper, aluminium, zinc, iron, platinum, tin, silver, and gold). Although there seemed to be no reason why this assumption should not be extended to all substances, it did not follow that ray-emission should necessarily be identified with radioactivity in the sense that it signified accompanying atomic change.

Piltschikoff (48), in 1906, recognized the so-called radiations which some metals produced as "Moser rays," named in honor of the physicist who discovered them (1). He found that these rays could be deflected by a stream of air, but not by a magnetic or electrical field.

In 1906 Russell (47) reported the results of new tests, which were confined largely to the action of plant substances on the photographic plate in the dark. Leaves, seeds, roots, bulbs, and, in fact, practically all vegetable substances acted in the same way. In many instances the tested material was subjected to considerable pressure before being brought in contact with the photographic plate in the dark. As a working hypothesis Russell reaffirmed the fact that the active material in these substances was hydrogen peroxide. He called attention to the fact that this action on the photographic plate by such substances is naturally what would be expected to occur, supposing, as has been stated by Usher and Priestley (37), that hydrogen peroxide and formaldehyde are the first products of plant growth.

Dombrowsky's dissertation (49), in 1908, covered the literature of this subject thoroughly. Glass, quartz, paraffin, silver, gold coin, platinum, and palladium were left in contact with a photographic plate for two days. No action was evident on development. On the other hand, zinc, aluminium, and cadmium, when left for a similar period in contact with the plate, produced dark spots on the sensitive film when the plate was developed. Bright spots on the metals caused very intensive darkening. Tests were also made to avoid direct contact by placing the metals from 2 to 5 mm. from the plate; in these the active substances also had an effect. When a metal sheet was caused to act on the photo-sensitive surface at different distances, the darkening of the plate decreased with the distance and became diffused. Various screens interposed between the active materials and the plate had different effects. Substances like celluloid, gelatin, and collodion permitted the transmission of the activity. Completely dried gelatin, however, was practically impermeable. Activity at higher temperatures was more intensive than that at lower temperatures. Dombrowsky's experiments showed that a metal sheet exposed to hydrogen peroxide vapor adsorbed a little of it and that there was a slow chemical reaction between the hydrogen peroxide and the silver bromide-gelatin film. The final blackening of the plate on development was the result of both of these processes. This investigator found that the metals alone were not active, except in the presence of moisture and oxygen.

Elizabeth Legrady (50), in 1908, was unable to obtain any darkening of the photographic plate by cadmium, copper, aluminium, magnesium, and zinc, when exposed in an atmosphere of hydrogen, nitrogen, oxygen, or air, either dry or moist. As a result of her tests she was led to the conclusion that these metals in themselves do not exert any action on the photographic plate, and do not form hydrogen peroxide or any radiation capable of producing an image. She considered the effect reported by others to be due to an ionizing action, the resultant image being produced by ionized hydrogen.

Saeland (51), in 1909, observed that if immediately after polishing, metals (Mg, Zn, Al, Pb) were placed in vacuum, dry air, or hydrogen, they caused practically no blackening on the photographic plate in the dark. His tests led him to conclude that such action is due entirely to the chemical action of the hydrogen peroxide formed, and therefore could not be due to a radiation.

In 1908, Russell (52) reported that the action of ordinary resin on the photographic plate was rather slow, exposure from two to three days being necessary to obtain a good picture. "Amber resin," however, was more active. Increasing the temperature augmented the activity, so that an exposure of only from three to four hours was necessary. Abietic acid, an important constituent of resin, gave a distinct image after exposure for eighteen hours. The presence of oxygen appeared to be necessary for this action. Russell believed that the action of resins and related bodies was produced by a vapor rather than by any form of radio-activity.

The work of O. and A. Dony-Henault in 1908 (53) convinced them that the photographic action of a solution of hydrogen peroxide on a silver bromide-gelatin plate was not due to radioactivity, but to the effect produced by the hydrogen peroxide existing as a vapor.

Brooks (54), in 1909 called attention to the fact that the substances tested by Russell and others owed their activity to their ability to form organic peroxides by autoxidation. Freer and Novy (26) and Clover and Richmond (27) had previously shown that organic peroxides were slowly hydrolyzed by water, with the formation of hydrogen peroxide.

Ebler (55), in 1909, explained the action of metals on the photographic plate on the supposition that the metal becomes oxidized by atmospheric oxygen, a higher oxide being formed (with carbon dioxide in the air), finally producing hydrogen peroxide, moisture also being present. He found that zinc and many other metals behaved in this manner. According to his conclusions, aside from the metallic surface, oxygen and a moist atmosphere were necessary to produce the effect observed.

For the next fourteen years, little work, if any, appears to have been done in this field. Rumpf (56), in 1923, from his experiments with zinc showed that its action on a photographic plate can not be due to a wave radiation or to electrons but must be due to a chemical reaction caused by oxidation by very small quantities of hydrogen peroxide.

The investigations of Sheppard and Wightman (57), in 1923, indicated that it is very unlikely that hydrogen peroxide acts as a reducing agent on silver halide. They believed that the silver halide was affected by the energy evolved primarily as a chemi-luminescence of short wave-length, this energy being the result of the decomposition of hydrogen peroxide which takes place in the presence of catalysts. The work of Wightman, Trivelli and Sheppard (60), in 1925, showed that acid hydrogen peroxide is slightly more effective than alkaline peroxide in producing an image.

Baughman and Jamieson (59) have shown that freshly expressed oils and fats possess no activity towards the photographic plate until they have been exposed to the sun for several hours. The saturated fatty acids were faintly active before exposure to the sun, becoming extremely active after exposure. Heating to approximately 120° for several hours appeared to destroy or weaken this activity. These results seemed to them to confirm Russell's view that the active substance is hydrogen peroxide.

Kugelmass and McQuarrie (61) came to the conclusion that ultraviolet light is not emitted by cod liver oil and certain other substances prescribed for rickets when they are oxidized in alkaline media. The darkening effect produced on the photographic plate by such substances is held to be due to reducing vapors, an effect comparable to that obtained by Russell and others with hydrogen peroxide.

Stutz, Nelson, and Schmutz (62) have recently found that a 0.01 per cent solution of hydrogen peroxide in water produces an image comparable to the effect produced by linseed oil after it had been exposed to the light for an hour. In the case of drying oils, it was indicated that a rapid reaction took place, confined chiefly to the surface, resulting in the formation of a skin which was relatively impervious to hydrogen peroxide. When this skin was broken, hydrogen peroxide was again actively evolved. They conceded that the primary oxidation of an oil was probably due to a molecular autoxidation, where molecules of oxygen were added at the double linkings, thus forming peroxides. Their work is being continued.

The foregoing résumé of the literature shows that the effect of various metals and organic substances upon photographic plates in the dark has been extensively studied since its discovery by Moser (1) in 1842, and that many explanations have been proposed to account for the effects. Some workers, especially the earlier ones, consider the phenomenon to be due to radiations of some sort. The majority of students, however, believe that they are produced by an emanation, hydrogen peroxide being most frequently considered as the active agent.

EXPERIMENTAL WORK

The principal part of the experimental work was done on plant substances and other organic material. A large variety of plant materials were tested. Some of them were used in the powdered form and others, such as leaves and flowers, after they had been placed in a plant press and then laid directly against the photographic emulsion. Empty desiccator jars covered with black paper and kept in a dark room were used for the tests. In some instances the material was placed directly on the photographic plate; in others it was placed in a shallow dish and the photographic plate was allowed to rest upon the dish, emulsion side down, with an interposed mask of cardboard or other inactive material containing some distinctive cut-out figure. The periods of exposure ranged from 2 to approximately 16 hours. Room temperature (22° to 24°) was usually maintained. After the test the photographic plate was developed and fixed according to the usual methods.

In order to make sure that the experimental procedure described was adapted for bringing out the phenomena under investigation, tests were made on some of the metals found by earlier workers to be active—zinc, magnesium, cadmium, and aluminium. All of these produced images on the plate, confirming the findings of previous workers.

TESTS ON PLANT MATERIAL

Many plant products affected the photographic film in the dark. Ground nutmeg produced a slight effect at a room temperature of 22° and a much more marked effect after an exposure for 16 hours at 46°. Ground cinnamon behaved similarly, a distinct picture being obtained after a 4-hour exposure at 45°. Grapefruit and orange skin gave an image only after being exposed to the plate for 16 hours at room temperature. Sandalwood (*Santalum album*) showed an effect after exposure for 2 hours at 45°, but practically none after an exposure for 16 hours at room temperature. Powdered aniseseed and cardamon berries gave no definite results.

Orange leaves, lemon leaves, orange blossoms, grapefruit leaves, oak leaves, and maple leaves were first dried by placing them between blotting paper and subjecting them to moderate pressure. At room and higher temperatures all gave an image which in the early stages of development of the plate was distinctly visible, but which was so weak that prolonged development, in an effort to make it intense enough for reproduction, actually resulted in its disappearance.

Certain plant materials were inactive toward the photographic plate in the dark at either 22° or 45° and previous exposure to sunlight for several hours did not appear to produce any activity in them. The following substances behaved in this manner: Lavender flowers, celery seed, coriander fruit, fennel fruit, ground mace, asafoetida, fenugreek, sassafras bark, camphor gum, ground mustard, turmeric (powdered), corn starch, rice starch, potato starch, wheat flour, rye flour, barley flour, and white corn meal.

TESTS ON OILS

Many of the essential oils and allied substances gave distinct images on the photographic plate. Lemon oil, oil of citronella, citral, oil of peppermint, and cedarwood oil gave distinct reactions after exposure for 16 hours at room temperature (24°). Pine oil and oil of eucalyptus showed no reaction after exposure for 16 hours at room temperature, but were distinctly active after an exposure for 4 hours at 50°, and 52°.

TESTS ON MISCELLANEOUS SUBSTANCES

Wood turpentine, abietic acid, and powdered rosin gave pictures on developing after an exposure for 16 hours to the photographic plate in the dark at 22°. Benzaldehyde also gave a distinct reaction under the same conditions. No reaction was obtained with the following substances, either before or after exposure to the sunlight for a considerable time: Menthol, vanillin, pyridine, nicotine, carbon bisulphide, ether, and naphthalene. It is interesting to note, in this connection, that aromatic principles gave the most striking results, particularly at 45° or slightly higher temperatures.

The pictures obtained with plant material strikingly resembled those obtained with hydrogen peroxide. Sheets of filter paper impregnated with titanium sulphate solution, which is often recommended for determining hydrogen peroxide, were placed over the tested material. Where these sheets rested on the edge of the petri-dish containing the active material they took on a yellow-orange color, suggesting that the active substance is actually hydrogen peroxide.

DISCUSSION

Some of the early workers assumed that the effects obtained on a photographic plate in the dark were due to radiations proceeding from the active substance. As early as 1857, however, Niepce de Saint Victor had recognized that the influence responsible for the darkening of the plate would not pass through glass, and more recently Russell and others have shown that sheets of mica and other minerals are impervious. If radiations were sent out from the tested substances, they should have been able to penetrate glass and mica. On the other hand, cardboard, black paper and gelatine, through which the plate is affected would be capable of transmitting volatile or vaporous substances. Moreover, no positive demonstration of the nature of the supposed radiations has ever been given. Accordingly, the alternative view that emanations from the active substances produce the observed effects seems more plausible. That emanations are actually responsible is clearly shown by the following experiments:

a. Russell, Vincent, and others, have proved conclusively that the effect in question would penetrate all sorts of porous materials but not a single impervious screen.

b. Thomson found that the interposition of an air-blast between the active material and the photographic plate caused the image to elongate, showing that the effect is due to a stream of vapor capable of being deflected mechanically.

c. The writer found that an increase of temperature intensified the results obtained with certain plant materials, such as sandalwood. Almost without exception, these are known to contain volatile aromatic principles, the evolution of which would be accelerated by heating.

d. Hydrogen peroxide, even in very dilute solutions, produced effects indistinguishable from those obtained with various active substances. Glass and mica, of course, are impervious to its passage, but actual experiments show that cardboard and black paper screens allow it to penetrate readily.

e. Tests with titanium sulfate paper made by Russell and others, including the writer, gave the same results with several of the active substances as with hydrogen peroxide.

f. As shown by Lengyel, Russell, and Dombrowsky, both oxygen and moisture must be present to produce a darkening of the plate. As hydrogen peroxide could not be produced in their absence, the view that this compound is the active agent seems inescapable.

g. Admittedly, these considerations do not prove that hydrogen peroxide is the only substance that is active. As has been suggested by Brooks, organic peroxides may also be present. However, it seems simpler to assume that hydrogen peroxide is the active substance in all cases.

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