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# Understanding the Chemistry of the Development of Latent Fingerprints by Superglue Fuming

**ABSTRACT:** Cyanoacrylate fuming is a widely used forensic tool for the development of latent fingerprints, however the mechanistic details of the reaction between the fingerprint residue and the cyanoacrylate vapor are not well understood. Here the polymerization of ethyl-cyanoacrylate vapor by sodium lactate or alanine solutions, two of the major components in fingerprint residue, has been examined by monitoring the time dependence of the mass uptake and resultant polymer molecular weight characteristics. This data provides insight into the molecular level actions in the efficient development of latent fingerprints by superglue fuming. The results show that the carboxylate moiety is the primary initiator of the polymerization process and that a basic environment inhibits chain termination while an acidic environment promotes it. The results also indicate that water cannot be the primary initiator in this forensic technique.

**KEYWORDS:** forensic science, cyanoacrylates, fingerprints, polymerization, initiation

A common forensic analytical tool is the fuming of cyanoacrylates (superglue) to develop latent fingerprints (1–3). The implementation of this technique involves the exposure of a latent fingerprint to ethyl-cyanoacrylate (ECA) vapor in an enclosed chamber. The specific mechanism by which this technique develops the fingerprint is that when the fingerprint comes in contact with the cyanoacrylate monomer in the vapor, white polymer grows along the ridges of the print, with virtually no polymer deposited on background areas (1,3). The ECA polymerizes on the ridges of the fingerprint to form micron size morphologies, such as the noodles or blobs that are shown in Fig. 1. These morphologies provide the optical contrast that is needed to visualize the fingerprint. The technique is known to be most effective when the latent print is on a non-porous substrate such as metals or plastics. Additionally, if the substrate on which the print lies is either white or transparent, secondary techniques can be employed to exude contrast (4).

In addition to the quality of the obtained print, the cyanoacrylate fuming method also reveals traces of blood and sweat that are exposed to the vapor (2). The cyanoacrylate will coat the droplets much the same way ECA is used to coat ice crystals (5,6). More important than the ability to reveal these traces of blood and sweat is the non-destructive nature of the ECA fuming method. A study using a variety of forensic PCR kits found that presence of the ECA did not prevent successful DNA testing of the blood or sweat (2).

Unfortunately, neither the mechanism of polymerization nor the molecular-level reason why polymer grows from the fingerprint ridges, but not between the ridges, is well understood. Nor is it well understood how to optimize this process. For instance, consider the two morphologies shown in Fig.1. These morphologies provide significantly different contrast in the resultant visual print, yet the molecular level actors that are responsible for the appearance of these two morphologies is still unclear.

It is assumed that some component of the fingerprint that resides on the ridge of the fingerprint serves as an initiator to the polymerization of the ECA to form a polymeric layer only in those spots that contain the initiator. Eccrine sweat, which makes up the fingerprint residue, has numerous components, many of which may initiate the polymerization of cyanoacrylates, including amino acids, water, and sodium lactate. In order to optimize the development of fingerprints in a broad range of conditions, it is necessary to determine the controlling molecular actors in this initiation and subsequent polymerization process.

Since its inception, there have developed, empirically, many methods by which ECA fuming can be used to develop latent prints. Early methods involved ambient temperatures and very long exposure times (3) but over time, the method has been refined to more efficiently obtain the highest quality prints. Currently, the method of choice for the fuming of latent prints using ECA involves the rapid heating of the superglue (1,3). This causes the glue to turn into a vapor, which then reacts with the fingerprint residue to grow polymer along the print ridges. Typically, prints are completely developed within 2 min (3).

In one effort to better understand the print development process, researchers have examined the chemical composition of fingerprints (7). The common adult fingerprint was found to be comprised predominately of an aqueous mixture of eccrine sweat (3,7,8). Eccrine sweat is secreted from the palms of the hands, soles of the feet as well as all other non-hairy surfaces (3,8). Although the exact composition of an individuals' eccrine sweat may vary slightly with lifestyle and diet (8), the general solute composition of eccrine sweat is presented in Table 1. These purely eccrine prints can however, become contaminated when the hands come into contact with other regions of the body (8). The hairy surfaces of the body have sebaceous glands buried deep within the hair follicles. These sebaceous glands secrete sebum, an oily mixture composed of fatty acids, triglycerides, and other components (3,8). Fortunately, sebum contamination was found, by weight, to be a minority component

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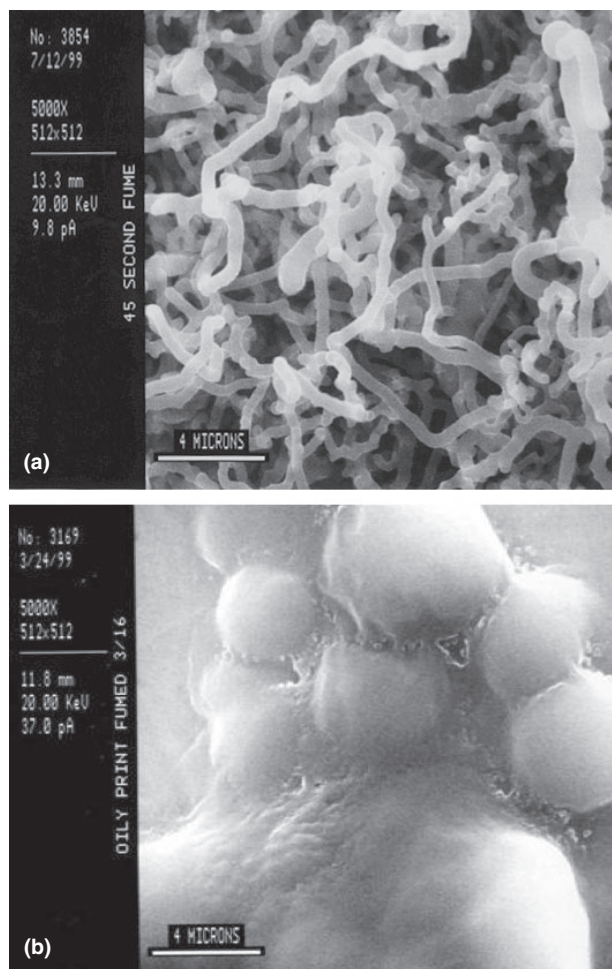[2]Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831.

FIG. 1—*(a) Cyanoacrylate polymer formed on the ridge of an eccrine sweat latent fingerprint. Eccrine fingerprints are deposited from clean hands containing little to no sebaceous material. (b) Cyanoacrylate polymer formed on the ridge of a latent fingerprint that has touched oily, sebaceous region of the body.*

TABLE 1—*The solute composition of eccrine sweat, the sweat secreted from the palms of an individuals hands.*

| Components of Eccrine Sweat | Abundance (wt%) |
|---|---|
| NaCl | 43.83 |
| Lactic acid | 29.22 |
| Urea | 11.69 |
| Amino acids | 7.79 |
| Others | 4.97 |
| $NaH_2PO_4$ | 1.75 |
| Glucose | 0.44 |
| $K_2PO_4$ | 0.31 |

of the total composition of a latent print and thus eccrine sweat remains the primary source for initiators of the ECA upon fuming. Lewis et al (3) found that the presence of sebum contamination did influence the aging of a fingerprint, but its presence was not required for print development. Therefore, sebum contamination will not be considered in our current investigation. Inspection of Table 1 indicates that the primary components of eccrine sweat are NaCl, lactate, and various amino acids. These three most abundant components then become preliminary suspects as initiators of ECA polymerization.

Unfortunately, there is very little literature available pertaining to the vapor phase polymerization of cyanoacrylates. Studies observing cyanoacrylate growth from snow flakes, ice droplets, and tobacco smoke utilize the technique but do not investigate the growth process (5,6). Therefore we must rely on solution-based chemistry to guide our understanding of the fuming process.

The polymerization of ECA in solution has been well documented (9–15). ECA, in the presence of a Lewis base, is known to polymerize via an anionic mechanism. An anionic polymerization consists of an anionic Lewis base initiator attacking a monomer. The negative charge is then transferred to the monomer and subsequently attacks another monomer. This process is propagated until one of two events occurs, either the monomer supply is exhausted or the anion will be terminated upon colliding with the terminating agent. If not terminated, the anion will remain as what is referred to as a living polymer, which retains the ability to propagate further should additional monomer be introduced to the system. In most cases, successful anionic polymerization requires the careful choice of initiator and the complete absence of terminating agents such as water, air, and especially acids.

While ECA does polymerize anionically, its unique chemical structure makes it unlike conventional anionic polymerizations. The chemical environment created by the presence of both the cyano and the acrylate group adjacent to the vinyl group allows for the formation of a very stable anion (10,12,16), when attacked by a Lewis base. Thus almost any Lewis base will initiate the anionic polymerization of the cyanoacrylates (9–11,17). In fact, even water has been shown to initiate the solution polymerization of ECA (10,16,18). One of the few Lewis bases that are not capable of initiating polymerization is the chloride ion (19). Thus, we can remove NaCl from our list of suspected initiators.

There are several other important features of solution polymerization of ECA that are relevant to the fuming process. For instance, it is known that the presence of acidic protons will affect the polymerization of ECA. Strong acids such as metaphosphoric acid (10), phosphorous pentoxide (10,17), and picric acid (10) are used commercially as stabilizers to prevent premature polymerization of superglue (13,16,19). The presence of weaker acids does not prevent polymerization but reduces the rate of chain growth and leads to lower molecular weights if sufficiently abundant (20,21). In other solution polymerizations, acids are used to terminate reactions through proton transfer (9,12). The role of the acid is of special interest in this investigation as in a previous study, the chemical environment (acidity/pH) of the print was shown to be extremely influential on the quality of the obtained print (3).

Thus, in order to effectively optimize this promising forensic technique, a more thorough understanding of the chemistry involved in the polymerization process that creates the visible morphology on the fingerprint residue is needed. In the present study, two model systems are studied for their ability to grow ECA polymer from the vapor phase. We seek to correlate the growth of polymer to specific initiator functionalities and chemical environment, in order to more fully understand the chemistry of the fuming process. One system that is studied contains the lactate ion, the second most abundant compound in eccrine sweat. The carboxylate functionality found in lactate has been used effectively, in the form of various acetate salts, to polymerize ECA in solution. The second system that is studied uses the simple amino acid alanine as the initiator. Alanine was chosen to represent the amino acids present in eccrine sweat because the R substituent group is a methyl group that will not act as an initiator. Alanine does however, have both the carboxylate and amine functionality capable of initiating polymerization.
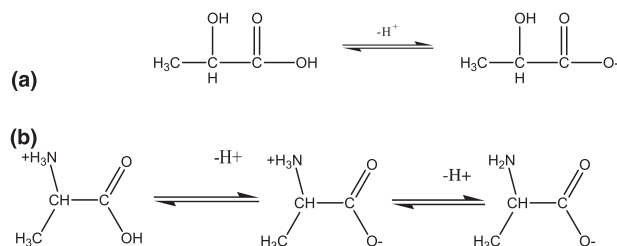
FIG. 2—*The structure of (a) a lactate molecule and (b) an amino acid, two of the major components of eccrine sweat, and the potential ionic states observed in solution.*

The influence of the pH of the two systems on its ability to polymerize ECA from the vapor is also examined. The pH was altered for several reasons, including the ease with which the pH could be varied, the fact that the method by which the pH is adjusted provides a mechanism to control which ions are introduced into the systems, and finally, because the pH has a direct effect on the electronic structure of the initiators that are shown in Fig. 2. In the lactate system the variation in pH dictates the counter ion at the carboxylate group, either a sodium or hydrogen atom. In the alanine system the carboxylate group is altered in much the same way as in the lactate system; however, pH adjustment also impacts the protonation of the amine as well as the zwitterionic nature of amino acids. Amino acids display zwitterionic behavior around the isoelectric point (6.02 for alanine).

## Method

The ability of these initiators in various electrical environments to initiate and polymerize ethyl cyanoacrylate vapors is investigated. To complete this, initiating solutions were prepared by adding 54.4 mg of either alanine (99%, Sigma-Aldrich, St. Louis, MO) or sodium lactate (99% Sigma-Aldrich) and distilled water in a 100 mL flask. This concentration was chosen to match the total solute concentration found in eccrine sweat. Adding either 6 M NaOH or 6 M HCl and reducing the amount of solvent to maintain a consistent overall concentration raised and lowered the pH of the solution respectively. To mimic the fuming method, these solutions were spotted on steel planchets and placed into ECA fumes. As a control, planchets that are dotted with distilled water were also fumed.

Prior to their use, stainless steel planchets were cleaned by sonication in toluene, a 1:1 water:ethanol solution, and distilled water. The planchets were then dried with an air hose, weighed and stored until needed. 45 μL of a solution were distributed on stainless steel planchets (radius *c.* 1.6 cm) in 3–5 μL droplets. The planchets with droplets, once prepared, were fumed within 30–60 sec after the initiator was deposited.

Sample fuming was completed by heating *c.* 2 g of ECA (Sirchie, Youngsville, NC) in an aluminum-weighing dish to a temperature of 150°C. Once white fumes began evolving from the dish, the system was enclosed in a chamber of volume 2500 cm³ with the stainless steel planchet suspended 5 cm above the vapor source. After the fuming process, the samples were allowed to dry at ambient conditions until constant mass (*c.* 24 h). Each sample was repeated ten times to ensure reproducibility.

To monitor the polymer growth, the mass of the polymer formed on the initiating surface was determined using mass by difference between the fumed and the unfumed planchets. Mass measurements were performed on a Mettler/Toledo AG245 microbalance with a sensitivity of 0.01 mg. To monitor the characteristics of the

polymer, the molecular weight and polydispersity of the poly(ethyl cyanoacrylate) samples was determined using a Waters 600E Gel Permeation Chromatography system with three Waters styragel columns (HR-1 [100 Å], HR-3 [$10^3$ Å], HR-5E [mixed bead]) or two Polymer Labratories PL gel 5 μm mixed D column and a Waters 410 RI detector. All GPC experiments samples were run in THF (Aldrich) as the eluting solvent and calibrated using polystyrene standards.

## Results and Discussion

The lactate ion contains a carboxylate functionality that can initiate the ECA and should therefore, if capable of initiating polymerization, provide an anionic system. Figure 3 shows the mass accumulation of cyanoacrylate on the lactate planchets as a function of time for various pH values as well as that of a planchet that contains only distilled water. The neutral lactate system is closest to the actual pH of eccrine sweat and the results are compared to that of the pure water. This comparison demonstrates that the lactate solution accumulates cyanoacrylate more rapidly than water alone; however the increase is not as significant as perhaps one may expect. When the pH of the system is altered, there is a change in this comparison, although the change is subtle. Lowering the pH causes little to no change in the mass accumulated relative to the neutral lactate system, except at long exposure times. Alternatively, increasing the pH causes an increase in cyanoacrylate accumulation, most notably at long exposure time. The two most interesting features of this graph are that the presence of initiator increases the amount of cyanoacrylate deposited, and that changes in pH only alter mass accumulation at long exposure times.

A comparison of the results for the lactate system to those of the alanine system will provide insight into the influence of the amine functionality as an initiator, as both initiators contain the carboxylate functionality. The time dependence of the mass accumulation in the alanine-initiated systems is presented in Fig. 4. The time dependence of the mass accumulation for the neutral pH alanine is very similar to that of the lactate system. The presence of the initiator increases the amount of cyanoacrylate accumulation relative to the pure water, confirming that the presence of either lactate or alanine ions is important to the success of the fuming process. Also, mass accumulation at short exposure times is consistent with that of the lactate system. Due to previous studies of amine initiated anionic polymerizations, it was suspected that the amine-initiated growth
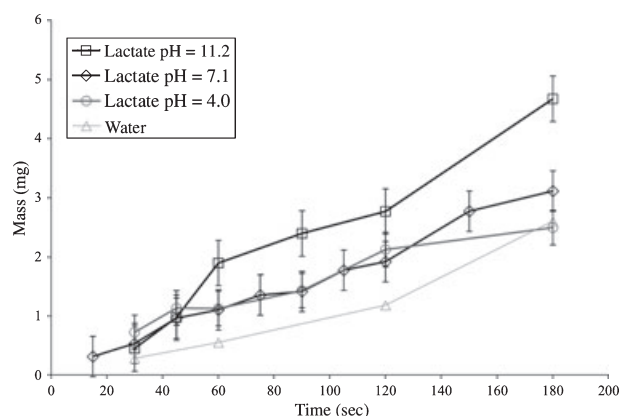


FIG. 3—*The mass of ethylcyanoacrylate accumulated in lactate-initiated systems during exposure to the monomer vapor.*
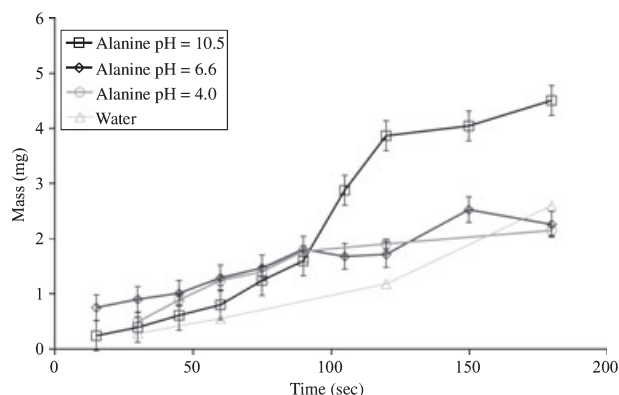
FIG. 4—*The mass of ethylcyanoacrylate accumulated in alanine-initiated systems during exposure to the monomer vapor.*

may occur at a slower rate than that of lactate, particularly at short exposure times (9,13,19,21).

Adjusting the pH of the alanine samples provides similar results to those observed for the lactate system. Lowering the pH appears to have no effect on the rate or amount of cyanoacrylate accumulated (even at long exposure times); while an increase in pH shows an acceleration of mass accumulated at long exposure time. Therefore, based on this data, both initiators investigated take up more ECA from the vapor with an increase in pH, particularly at longer exposure times. Furthermore the mass uptake in both systems is nearly identical, suggesting that the alanine system initiates via the same mechanism as the lactate, i.e., by the carboxylate functionality.

This data provides insight into the uptake of the amount of monomer, but little insight into the actual polymerization process. For this perspective, the molecular weight characteristics of the accumulated material on the steel planchets are needed. Figure 5 shows the number average molecular weight of the accumulated material on the lactate planchets as a function of fuming time. In this analysis, there is a stark difference between the samples accumulated on the neutral lactate compared with pure water. Although pure water was shown to accumulate similar mass to the neutral lactate system, the water grew only very few chains of any significant length, essentially producing only oligomers of no significant molecular weight. This very clearly indicates that lactate ions are
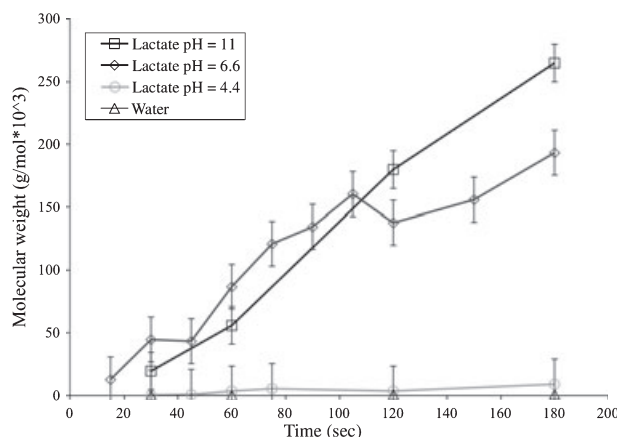
responsible for some of the initiation of the ECA during the fuming process.

Moreover, it provides direct evidence that water is not the initiator of the cyanoacrylate polymerization during fuming. Water is often thought to be the primary initiator of the cyanoacrylate in superglue fuming by forensic investigators (22,23). However, this data unequivocally proves that water does not create the long chain polymers that are observed in cyanoacrylate developed fingerprints.

Examining the change in the molecular weight of the formed polymers also provides insight into the effects of changing pH on the polymerization process. Lowering the pH limits polymer growth to short chains of very low molecular weight, oligomers, similar to what is observed for pure water, while increasing the pH significantly increases the molecular weight of the resultant polymers relative to that of the neutral lactate system.

Lowering the pH can potentially influence the polymerization process by three different mechanisms, the lower pH may prevent initiation of the cyanoacrylate, it may slow the rate of propagation, or it may increase the rate of termination. In solution studies, an acidic environment does not prevent initiation but does repress propagation (20) and cause termination (21). For the higher pH sample, the molecular weight follows the same trend as the mass accumulation analysis, creating larger molecular weight polymers but only at long exposure times. The higher pH may impact the polymerization process by providing additional $OH^-$ initiators, but this does not appear to be the case since the molecular weight only varies at long exposure time and the increase in initiators would affect the entire process. The deviation at long exposure times suggests that the influence of pH occurs at the latter stages of polymerization rather than initiation.

Further insight into the role of the pH on the fuming process can be gained by examining the change in molecular weight of the alanine-initiated polymers as a function of fuming time (Fig. 6), where a trend is observed similar to that of the lactate system. The similarity of the evolution of the molecular weight of the polymer for the lactate and alanine system again suggests that similar polymerization processes govern the two systems. More precisely, the short exposure times exhibit no indication of the slow-initiation kinetics expected for amine initiated systems (9,13,19,21). Also, the long time molecular weights for the neutral alanine are nearly the same as the lactate system. There is no indication of larger overall molecular weights expected for amine initiated systems, and certainly no evidence of the extremely large molecular weights expected in the presence of zwitterionic growth (11,19).
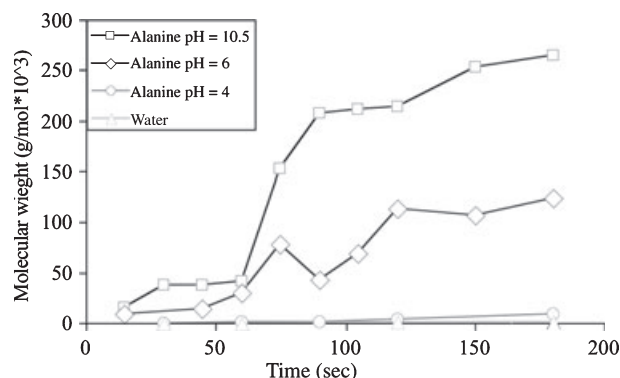


FIG. 5—*The molecular weight of ethylcyanoacrylate in lactate-initiated systems plotted as a function of exposure time at various pH's.*



FIG. 6—*The molecular weight of ethylcyanoacrylate in alanine-initiated systems plotted as a function of exposure time at various pH's.*

Adjustments to the pH further support the similarity of the lactate and the alanine initiated systems. Lowering the pH essentially eliminates the growth of any substantial molecular weight polymers just as was observed in the lactate samples. Similarly, when the pH is increased, it is only at longer exposure times that the high pH became distinguishable from the neutral pH. The higher pH continues to grow larger polymers while the neutral system appears to level off. Through this direct comparison of lactate and alanine, it is obvious that the polymerization processes indeed occur similarly in the two systems. The combination of all these observations leads to the conclusion that the carboxylate group is the primary initiating site for the polymerization of cyanoacrylates in the fuming of superglue. Further studies in our laboratory have verified that the carboxylate moiety is a better initiator of ECA fumes than a primary amine (24).

It is also useful to understand the role of pH on the polymerization processes. For instance, it has long been known, empirically, that the addition of ammonia to the superglue fuming chamber improves the quality of the resultant prints (1). Is this related to the pH of the system? If so, how? Clearly, from the presented data, the presence of free protons in the acidic environment inhibits chain growth in some way. As mentioned earlier, the acidic environment may inhibit initiation or may serve as terminating agents. These two possibilities may be differentiated by determining the number of polymer chains that are formed as a function of pH. If the acidic environment is inhibiting initiation, there should be fewer chains present in the low pH sample than those of the neutral or basic environments. This is just a matter of accounting, where the total number of possible initiators can be calculated using (Eq. 1) by accounting for the volume and concentration of sample on the planchet

$$I_p = [I] * V * N_A \qquad (1)$$

where $I_p$ is the total number of initiators, which also equals the total number of potential chains, $[I]$ is the concentration of initiators in the solution, $V$ is the volume of the sample dotted on the planchets and $N_A$ is Avogadro's number. Moreover, this can then be compared to the actual number of chains that are formed on the planchet as determined by (Eq. 2),

$$N_{ch} = m * N_A / M_n \qquad (2)$$

where $N_{ch}$ is the actual number of chains formed, $m$ is the mass of the polymer formed, $N_A$ is again Avogadro's number, and $M_n$ is the number average molecular weight of the polymer. These two values, the number of initiators and number of chains formed for the lactate samples are shown in Fig. 7, while the same data for the alanine samples are shown in Fig. 8.

The most striking feature of these figures is that initiation is not only suppressed in the low pH system, but in fact the low pH samples create the greatest number of chains. This provides critical data to picture the molecular level impact of changing the pH on the polymerization of cyanoacrylate. All initiating systems create a large number of polymer chains, however, the low pH generates the largest number of chains indicating that these systems have a greater occurrence of termination than the other systems. As a growing polymer chain is terminated, further incoming monomer must re-explore the surface to find a viable initiation site, thus leading to a greater number of chains. The increase of termination at low pH also explains the difference of the molecular weight variation with pH.

At long exposure times, the high pH systems continued to grow larger polymers while the molecular weight of the neutral system
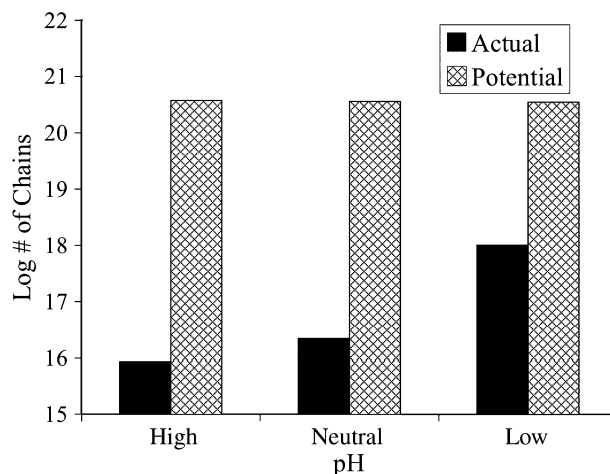


FIG. 7—A comparison of the number of potential lactate initiating groups to the number of actual polymer chains grown.
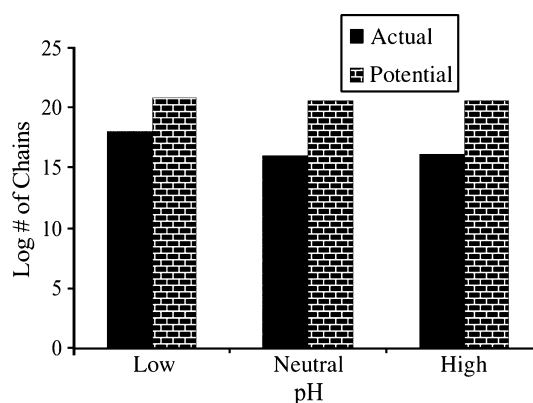


FIG. 8—A comparison of the number of potential alanine initiating groups to the number of actual polymer chains grown.

levels off. When termination occurs at these pH values (at a much lower rate than the low pH) it is most likely at long exposure time. At these later times, the polymer film is thick and the initiating surface is less accessible, making chain regeneration difficult. Thus, these results also indicate that there is less polymer termination at high pH in a more basic environment. This information also agrees with the empirical evidence that addition of ammonia to a fuming chamber will improve the quality of the fingerprint, as our results suggest that it is this basic environment that is needed to successfully polymerize long chain molecules that are needed for the visual contrast.

## Conclusions

Using mass accumulation studies and molecular weight analysis, the primary components of eccrine sweat (lactate and alanine) are shown to be capable of initiating polymerization of ethyl cyanoacrylate. Both initiators grow similar amounts and molecular weights of the polymer. There was no indication of amine-initiated growth from the alanine system, which would have been characterized by slower initiation and larger overall molecular weights. The similarity in the data supports that both initiators grow the polymer by the same mechanism, initiation by the carboxylate functionality. The apparent carboxylate initiated growth in the alanine system

also indicates that in the presence of both amine and carboxylate ions, cyanoacrylate growth will preferentially occur with the carboxylate group. Comparison of these systems to that of pure water also indicates that water is not the initiator responsible for polymer chain growth in superglue fuming.

By varying the pH, the polymerization process is either suppressed or enhanced. Lowering the pH did not prevent cyanoacrylate from accumulating at initiating surfaces but did result in significantly lower molecular weights. Increasing the pH increases the overall mass accumulation as well as increasing the molecular weights. These changes however did not occur until longer exposure times. These results indicate that this is a result of the relative concentration of hydrogen ions, which act as terminating agents. Increasing the pH, or effectively lowering the concentration of hydrogen ions, reduces termination allowing for larger polymer growth.

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