Adsorption and Reduction in Bioactivity of Polychlorinated Biphenyl (Aroclor 1254) to Redroot Pigweed by Soil Organic Matter and Montmorillonite Clay[1]

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ABSTRACT

Aroclor 1254 polychlorinated biphenyl (PCB) applied to untreated (1.4% organic matter) and H_2O_2-treated (0.2% organic matter) Lakeland sand at rates of 0, 50, 100, 150, and 200 ppm caused significant reductions in redroot pigweed (*Amaranthus retroflexus* L.) growth at the high rates of application. Polychlorinated biphenyl decreased plant height in the H_2O_2-treated soil much more than in the untreated soil. A greater decrease in dry weight also resulted in the H_2O_2-treated soil, but only at the 200 ppm rate. In one study, soil was amended with montmorillonite clay and organic matter (peaty muck) at rates of 0, 2.5, 5.0, and 10.0% by weight and treated with 200 ppm PCB. The organic matter proved to be much more effective than montmorillonite in reducing PCB toxicity to pigweed at the lower rates of addition (2.5 and 5.0%), but no differences were noted between the two adsorbents at the 10% rate. Adsorption of ^{14}C-labeled PCB closely resembling Aroclor 1254 by the untreated and H_2O_2-treated soils, montmorillonite clay, organic matter, and activated carbon was measured. The order of highest to lowest adsorption was as follows: activated carbon $\gg$ organic matter > montmorillonite clay > Lakeland sand > H_2O_2-treated Lakeland sand. These experiments suggest that organic matter content of a soil, and clay content to a lesser extent, are involved in reducing the availability of soil-applied PCB to pigweed. Differences in reduction of PCB toxicity to pigweed are explained by differences in adsorption by the adsorbents.

Additional Index Words: **activated carbon, H_2O_2 treatment, PCB phytotoxicity, PCB inactivation.**

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Polychlorinated biphenyls (PCBs) are a ubiquitous pollutant found in terrestrial as well as aquatic systems (Gustafson, 1970; Nisbet and Sarofim, 1972; Harvey and Steinhauer, 1975). Agricultural lands may become a source of PCB entry into food crops following the application of contaminated sludges used as fertilizer (Lawrence and Tosine, 1976; Pahren et al., 1979; Strek et al., 1981). Levels of PCBs taken up by crops have generally been low (Strek and Weber, 1981), but accumulation of PCBs in soil may cause problems of increased toxicity and uptake.

Application of PCBs to soil has detrimentally affected the growth of soybeans and beets, but not that of corn and sorghum (Weber and Mrozek, 1979; Strek et al., 1981). Toxicity and uptake of PCB by crop plants and weeds were alleviated by application of activated carbon to PCB-treated soil. Polychlorinated biphenyls are reported to possess a high affinity for soil constituents, and movement in soils depends particularly on the clay and organic matter contents of soils (Strek and Weber, 1981). The bioavailability of low water-soluble, nonionic pesticides from the soil is primarily dependent upon organic matter content (Weber and Weed, 1974). For example, phytotoxic rates of trifluralin applied to 10 soils varied directly with the organic matter contents of the soils (Harrison et al., 1976). Polychlorinated biphenyls would be expected to behave similarly due to their nonionic nature, low water solubility, and demonstrated affinity for activated carbon.

The adsorption of PCBs from aqueous solution by soils has been demonstrated (Haque et al., 1974). Soil organic matter content seems to be the most important factor influencing PCB adsorption by soils (Haque et al., 1974; Haque and Schmedding, 1976; Scharpenseel et al., 1977a, 1977b; Lee et al., 1979), although specific surface area of all soil colloids may also exert considerable influence (Hiraizumi et al., 1979). The desorption of PCBs which may give some indication of their biological availability in soil has been reported to be relatively negligible (Haque et al., 1974; Musty and Nickless, 1974; Steen et al., 1978; Hiraizumi et al., 1979; Wildish et al., 1980). The mechanism of PCB adsorption in soils has been suggested to be physical (Lee et al., 1979).

The purpose of the research reported herein is to determine the influence of natural and added organic matter, as well as montmorillonite clay, upon the toxicity of soil-applied Aroclor 1254 to redroot pigweed and adsorption of the chemical by these adsorbents.

MATERIALS AND METHODS

Plant Studies

Soil used in the experiments was taken from the 0- to 15-cm depth of the A horizon of a virgin Lakeland sand (Typic Quartzipsamments; siliceous, thermic, coated; pH 4.0; cation exchange capacity: 1.5 meq/100 g; 1.4% organic matter, 5% clay, and 6% silt) obtained from a Johnston County, North Carolina site which had not been subjected to agricultural practices for at least 40 years (Table 1). The soil was air-dried and sieved through a 0.5-cm screen prior to potting. Plants were grown in 186-cm^3 styrofoam pots containing 150 g of soil limed to approximately pH 6.0 with $CaCO_3$. A portion of this soil was treated with hydrogen peroxide (H_2O_2) to remove the organic matter (Jackson,

Table 1—Properties of soils and adsorbents.

Adsorbent	Organic matter†	Specific surface area‡	A_{50}§	Relative ranking of adsorptivity
	%	m²/g	g	
Activated carbon	100	452.18	--	--
Peaty muck organic matter	92.2	1.23	0.0025	132
Montmorillonite	1.6	62.97	0.0210	16
Lakeland sand	1.4	0.41	0.0800	4
H_2O_2-treated Lakeland sand	0.2	0.62	0.3300	1

† Determined by ashing at 400°C.
‡ Determined by N_2 adsorption (Quantachrome Quantasorb Surface Area Analyzer).
§ Amount of adsorbent necessary for 50% adsorption of PCB using equilibrium concentrations of 66.80 to 62.61 ppb.

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1958) and potted separately. A peaty muck (Histosol, pH 3.4), obtained from near Pantego, N. C., and a Ca-saturated montmorillonite clay (Volclay from Panther Creek, Miss.,) were each added separately to untreated Lakeland soil in increments amounting to 0, 2.5, 5, and 10% of the total weight and then thoroughly mixed with the soil.

Analytical-grade PCB (Aroclor 1254), obtained from the Food and Drug Protection Division Laboratory of the North Carolina Department of Agriculture (Lot AM 51, originally obtained from Monsanto Co. in 1971), was dissolved in pesticide-grade hexane to make the stock solution. Twenty-milliliter aliquots of the stock solution were thoroughly mixed with untreated soil and H_2O_2-treated soil at rates of 0, 50, 100, 150, and 200 ppm, and the pots were equilibrated for 3 days to allow for solvent evaporation. A preliminary study showed that 200 ppm caused approximately 50% growth inhibition of pigweed in the Lakeland soil. Soil treatments to which organic matter and clay had been added were treated with 0 and 200 ppm rates of PCB.

Pots were planted to *Amaranthus retroflexus* L. (redroot pigweed) and placed in controlled environment chambers (200 hlx, 16-hour day, 30 ± 3°C). The soil was maintained at approximately 80% field capacity and fertilized as needed with a modified Hoagland's solution (Weber, 1977). The plants were grown for 4 weeks, after which height, fresh weight, and dry weight measurements were taken.

Adsorption Studies

Untreated and H_2O_2-treated soils, organic matter, and montmorillonite clay as described previously were used as adsorbents (Table 1). In addition, activated carbon (Nuchar SA, Westvaco, Inc.) was also included. A ^{14}C-ring-labeled PCB mixture resembling Aroclor 1254 [specific activity = 31.3 mCi/mmol, obtained from New England Nuclear (Lot no. 872-193), average molecular weight = 326.25] was used for the studies. The labeled material was an isomeric mixture of ^{14}C(U)-PCBs, approximately 54% chlorine by weight, having a greater Cl/biphenyl range than Aroclor 1254. An aqueous solution of PCB was prepared by adding 10-ml of ^{14}C-PCB (0.1 mCi) to approximately 2 liters of distilled deionized water in a glass vessel on a magnetic stirrer. Two-milliliter aliquots of each sample were added to a toluene-based cocktail (16.5 g 2,4-diphenyloxazole, 0.5 g 1,4-bis [2-(4-methyl-5-phenyloxazolyl)] benzene, 1,000 ml Triton X-100, and 2,000 ml toluene) and radioassayed in a liquid scintillation spectrophotometer. Counting time was 20 min or until 100,000 counts were reached, and counting efficiency ranged from 83 to 89%. Measured radioactivity was corrected for background activity and converted to dpm. The amount of activity in solution reached a steady amount after 1 week with minor decreases over time of < 2% per week. Concentration of the PCB in the stock solution ranged from 66.80 to 62.61 ppb during the course of the experiments with one exception. In adsorption studies performed with activated carbon, the concentration was 55.55 ppb.

Polychlorinated biphenyls are reported to have a great affinity for glass and plastic surfaces (Zitko, 1970; Gresshoff et al., 1977; Lee et al., 1979; Picer et al., 1979) and we also found this to be the case. The process of shaking 20-ml ^{14}C-PCB standards reduced the amount of ^{14}C-activity in aqueous solution to an equilibrium level of approximately 58% after 1 hour's time. ^{14}C-PCB which left the solution phase was assumed to have adsorbed to the glass surfaces inside the vessels. Adsorption studies reached equilibrium after 2 hours, and only a 3% decrease in recovery was noted between 2 and 6 hours of shaking; thus, a 4-hour shaking time was used. Adsorbents (air dried), in amounts ranging from 0.001 to 1.000 g, were added to 50-ml glass bottles containing 20 ml of aqueous PCB stock solutions. ^{14}C-PCB standards containing no adsorbent were used to estimate the amount of PCB available for adsorption (that not adsorbed by the glass) and recovery averaged 58.1 ± 9.0%. The amounts of PCB adsorbed were obtained by subtracting the equilibrium concentrations from the concentrations of the standards. Duplicate samples were used in all cases.

Statistical Treatment

Analyses of variance were performed on the data using various models for direct and interaction effects. Student's *t* tests between least squares mean (LS mean) pairs were used to determine the significance of differences. Linear regressions were fitted to experimental data and the equations which best fit the data were used to describe the responses. All effects are reported for significance at $\alpha = 5\%$ unless otherwise noted.

RESULTS AND DISCUSSION

Plant Studies

Treating the Lakeland sand with H_2O_2 for removal of organic matter substantially decreased its effectiveness in reducing the toxicity of the Aroclor 1254 (PCB) (Fig. 1a and 1b). The differences in plant growth attributable to H_2O_2 treatment was significant. Despite the small absolute difference in organic matter content between the two soils (circa 1.2%, see Table 1), an apparent difference in PCB toxicity did occur. The organic matter × PCB rate interaction effect was significant only for plant height, signifying that the re-

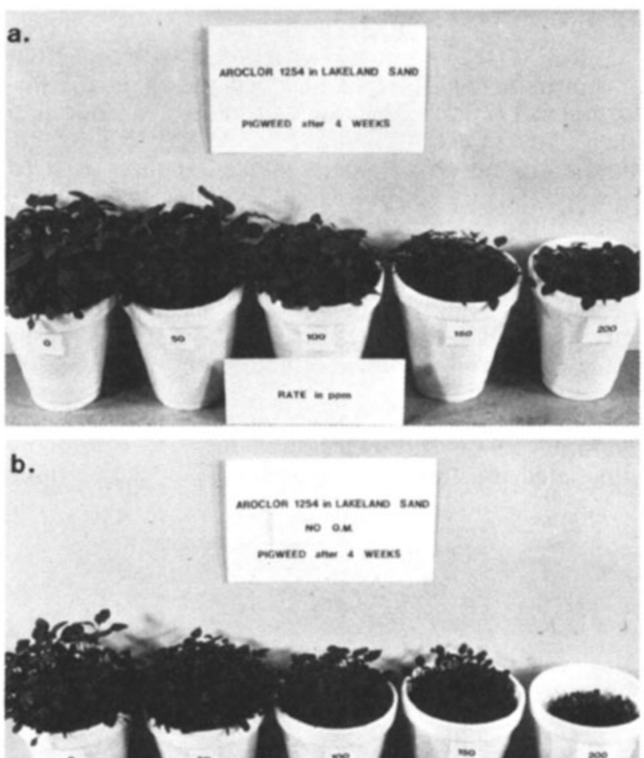

Fig. 1—Redroot pigweed growth on an (*a*) untreated and (*b*) H_2O_2-treated Lakeland sand containing 0, 50, 100, 150, and 200 ppm Aroclor 1254 (PCB).

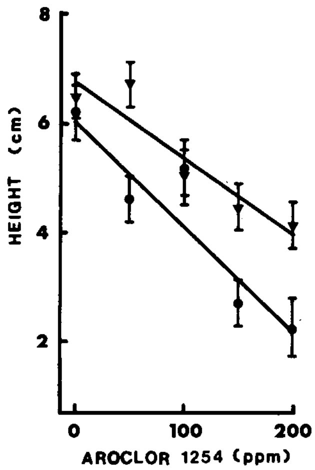

Fig. 2—Inhibition of pigweed height by Aroclor 1254 (PCB) applied to untreated (▼) and H_2O_2-treated (●) Lakeland sand. Standard error bars are included.

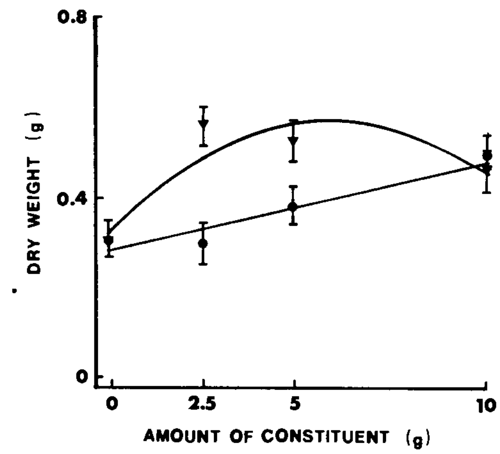

Fig. 4—Influence of montmorillonite clay (●) and organic matter (▼) amendments on toxicity of 200 ppm soil-applied Aroclor 1254 (PCB) to redroot pigweed. Standard error bars are included.

sponse of plant height to PCB rate differed between soil treatments (Fig. 2). The slope of the plant height vs. PCB rate response curve was considerably less for the untreated soil than for the H_2O_2-treated soil, indicating greater toxicity at each PCB rate in the soil with the organic matter removed. The PCB caused significant reduction in height at rates > 50 ppm in the H_2O_2-treated soil and at rates > 100 ppm in the untreated soil. Significant differences between LS means between the two soil treatments occurred at the 50, 150, and 200 ppm PCB rates.

Since the 200 ppm rate resulted in growth reduction of approximately 50% to pigweed grown in the untreated soil (Fig. 2), this rate was chosen to determine the relative effect of montmorillonite clay and organic matter on the phytotoxicity of soil-applied PCB to pigweed (Fig. 3a and 3b). Organic matter amendments reduced the deleterious effect of PCB much more effectively than did equal amounts of clay amendments. The responses of plant height, fresh weight and dry weight were almost identical to that of dry weight, so only dry weight data are included (Fig. 4). Increases in pigweed dry weights were noted following addition of both constituents. The dry weight increase proceeded in a quadratic fashion for the organic matter addition, where the lowest rate of 2.5% essentially eliminated the toxic action of the PCB. At all three rates of organic matter addition, pigweed dry weights were significantly higher than the control, which received PCB but no organic matter or clay. In pots receiving no PCB, the dry weight LS means for the control, 10% organic matter amended, and 10% montmorillonite clay amended were 0.601, 0.516, and 0.727 g, respectively. Significant differences in dry weights between the clay and organic matter treatments occurred at both the 2.5 and 5.0% rates of amendment. The type of colloid × amount of colloid interaction effect was also significant, as would be expected. At the 10% rate of addition, the safening effect of the clay approached that afforded by the organic matter. However, the dry weight LS mean of the 10% clay treatment receiving PCB was slightly but significantly lower than the 10% clay treatment receiving no PCB. No significant dry weight reduction occurred for the corresponding PCB-treated plants at the 10% organic matter addition. Although the affinity of PCB is apparently greater for organic matter than for montmorillonite, affinity to clay is strong enough to allow a corresponding reduction in toxicity at higher rates.

Adsorption Studies

In an attempt to explain differential reductions in PCB toxicity due to addition of clay or organic matter to the soil, the two soil treatments and three constit-

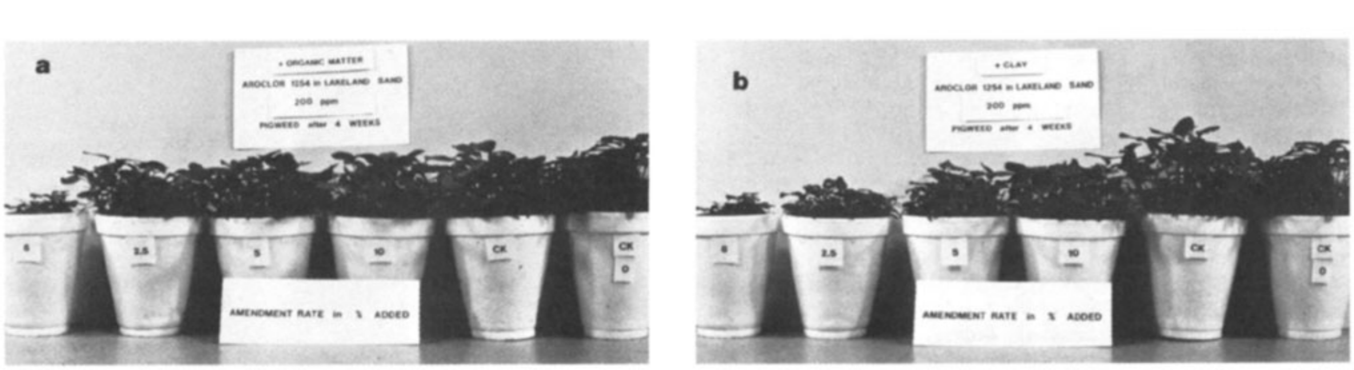

Fig. 3—Redroot pigweed growth on a Lakeland sand containing 200 ppm Aroclor 1254 (PCB) amended with 0, 2.5, 5, and 10% (*a*) organic matter and (*b*) montmorillonite clay by weight. Two controls contained no PCB and 0 (CK) and 10% (CK-0) of the respective amendments.

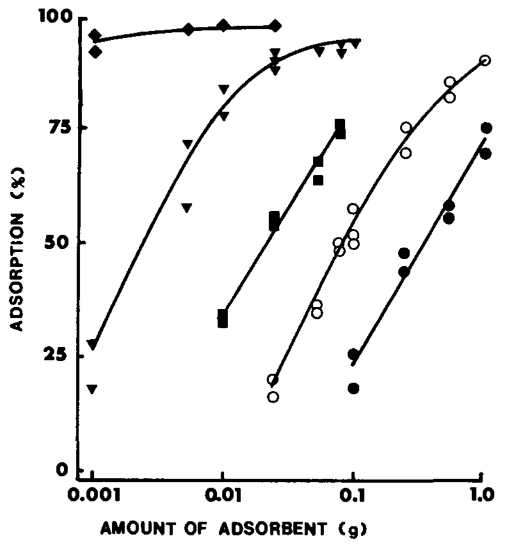

Fig. 5—Adsorption of ^{14}C-PCB from aqueous solution by activated carbon (♦), organic matter (▼), montmorillonite clay (■), Lakeland sand (○), and H_2O_2-treated Lakeland sand (●). (Amount of adsorbent in logarithmic scale).

uents employed for these experiments were used in aqueous adsorption studies with ^{14}C-PCB, which closely resembled Aroclor 1254. Relative adsorption was reported as the percent adsorption of available PCB in solution (as measured in standards) vs. the weight of adsorbent (log scale) (Fig. 5). As was expected, differences between adsorbents were highly significant, as were increases in adsorption due to amount added. The relative position of the curves indicates that activated carbon was most effective in PCB adsorption, followed by organic matter, montmorillonite, untreated Lakeland sand, and finally the H_2O_2-treated Lakeland sand.

To develop a ranking of the relative effectiveness of each adsorbent for PCB, it was decided to calculate the amounts needed to adsorb 50% of the available PCB from solution (Table 1). Activated carbon so efficiently adsorbed PCB that adsorption never went below 92%, so it could not quantitatively be compared with the rest (Fig. 5). The use of the 50% value for the remaining adsorbents is validated by the observation that the plots (Fig. 5) are almost parallel at that point, which means that the slopes are similar, within a certain range. The increase in adsorptive power, on a weight basis, of the untreated sand vs. the H_2O_2-treated sand was over fourfold (Table 1). In other words, the loss of a component comprising only 1.2% of the total soil weight invoked a 400% loss in PCB adsorbing capacity of that soil as a whole. Organic matter, with a relative ranking of 132, was eight times more effective in adsorbing PCB from solution than was montmorillonite with a relative ranking of 16. Lee et al. (1979) concluded that PCBs do not adsorb to the interlayer space of montmorillonite clay, thus only the exterior surfaces are probably available for PCB adsorption. By contrast, organic matter contains many interstitial spaces capable of adsorbing lipophilic compounds like PCB (Weber and Weed, 1974). Hiraizumi and co-workers (1979) reported that the specific surface area of adsorbents as measured with N_2 was a good indicator of PCB retention by adsorbents. The measurement does reflect the high adsorptivity of PCB by activated carbon and montmorillonite, but falls short for organic matter (Table 1). In addition, PCB adsorption by the untreated Lakeland soil was four times that for the H_2O_2-treated soil and yet specific surface area, as measured by N_2, was actually higher for the latter. It is apparent that the type of surface is as important as the amount of surface in estimating PCB adsorption. The increased adsorptivity of activated carbon over organic matter can probably be related to a tremendous increase in surface area of the former and possibly to a decrease in hydrophilic functional groups. These data suggest that N_2 surface area measurements probably do not measure relative adsorptive sites for PCB adsorption. Activated carbon was by far the most effective adsorbent for PCB, probably explaining the great reductions in phytotoxicity and uptake of ^{14}C-PCB by plants (Weber and Mrozek, 1979; Strek et al., 1981) and ^{14}C-PCB bioaccumulation by goldfish (Shea et al., 1980) observed by other investigators.

The difference in adsorptive capacity for PCB between the H_2O_2-treated (A_{50} = 0.3300 g) and untreated sand (A_{50} = 0.0800 g) was probably the major reason for the difference in plant heights obtained from the PCB bioassay involving the two treatments (Table 1 and Fig. 2). Similarly, differences between the adsorptive capacities of organic matter (A_{50} = 0.0025 g) and clay (A_{50} = 0.0210 g) may account for the different responses of pigweed dry plant weight produced in PCB-treated soil treatments (Table 1 and Fig. 4). These data suggest that reductions in PCB toxicity to pigweed were related to increased adsorption by various adsorbents.

Activated carbon and organic matter probably possess numerous lipophilic sites from which the PCB could not be easily replaced by water molecules or plant root exudates. Higher chlorinated biphenyls are reported to possess a higher affinity for soils than lower chlorinated biphenyls (Haque and Schmedding, 1976). Recent work showed that steric configuration of hexachlorocyclohexane (α,β,γ isomers) had a large influence on its adsorption to soils (Wahid and Sethunathan, 1979). Adsorption of the isomers was highly correlated to organic matter content of the soils. However, following oxidation of organic matter, adsorption varied between the isomers on the same soils. This suggests involvement of chlorines in adsorption to mineral surfaces by perhaps electrostatic attraction.

Montmorillonite clay was only one-eighth as effective in binding PCB as organic matter but it effectively reduced PCB phytotoxicity to the pigweed. Polychlorinated biphenyl adsorption to the clay surfaces occurred despite the hydrophilic nature of the surface, probably because of the very low water solubility of the compound and in a physical manner similar to PCB retention by the walls of the glass vessels in standards used in the adsorption studies.

CONCLUSIONS

Natural organic matter appears to be relatively effective in reducing the bioavailability of Aroclor 1254 (PCB) to redroot pigweed. The capacity of an untreated Lakeland sand (1.4% organic matter) to adsorb PCB from aqueous solution was approximately four times greater than that for H_2O_2-treated Lakeland sand (0.2% organic matter). This suggests that natural organic matter is relatively effective in decreasing the bioavailability of PCBs. Organic matter was over eight times more effective in adsorbing PCB from aqueous solution than was montmorillonite clay. This suggests that organic matter is much more effective than montmorillonite for biologically inactivating PCB. Activated carbon proved to be the most effective adsorbent of PCB from aqueous solution, which helps to explain previously observed reductions in PCB phytotoxicity and uptake following amendment of PCB-contaminated soil with carbon.

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