REVIEW ARTICLES

Recent development of organic electron transport materials*

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Abstract   This article reviews the recent development of organic electron transport materials applied in the fields of organic photoconductors, light-emitting diodes, field-effect transistors and solar cells. Several technologies for charge carrier mobility measurement are summarized and compared, and a series of basic principles for designing high-performance organic electron transport materials are suggested as well.

Keywords: electron transport, charge carrier mobility, organic semiconductors.

The organic charge transport material (OCTM) is a new class of organic semiconductive materials in which charge carriers (electrons or holes) injected can be transferred orderly under the electric field thus realizing charge transport. Relative to inorganic materials, OCTMs have many advantages such as low cost, non-toxicity, ease of fabrication and feasibility to satisfy various needs through chemical modification, especially the potential of making soft devices. Today they have been widely applied in the fields of xerography[1,2], chemical sensor, light-emitting diodes (LED), field-effect transistor (FET), solar cells and so on[3~6].

OCTMs include organic hole transport materials (OHTMs) (p-type) and organic electron transport materials (OETMs) (n-type). Up to date, most of the OCTMs reported are OHTMs[7], because of the OETMs' difficulty in synthesis and characterization. So it is urgent to summarize the study on OETMs and bring forward some principles for material design in order to promote the development of OETMs.

1 Development of OETMs

The development of OETMs is much slower than that of OHTMs. Tris (8-quinolinolato) aluminum (Alq3)[8] and 2-(4-diphenylyl)-5-(4-tert-butylphenyl)-1,3,4-oxa diazole (PBD)[9] were investigated earlier and have been reported in details,

which are widely used in organic and polymer LED devices. But easy crystallization and poor compatibility limit their further development. In the recent years, due to the consideration of environmental protection and unbalanced development between p-type and n-type semiconductor, more and more attention has been paid to OETMs. Up to date, according to reports[10], OETMs can be classified into the following sorts: planar extended π-system, polymer electron transport and other compounds.

1.1 Planar extended π-system materials

1.1.1 Metallophthalocyanines

Metallophthalocyanines have demonstrated remarkable semiconducting properties. They have been widely studied as photoconductors, solar cells and sensors. However, the charge transport properties of these compounds have received less attention presumably because of their low field-effect mobilities [cà. 10^{-4} cm^2/(V·s) reported], e.g. in the case of phthalocyanine nickel (NiPc)[11~13].

Guillaud et al.[11] found that lutetium bisphthalocyanine (LuPc$_2$) and thulium bisphthalocyanine (TmPc$_2$) exhibited field-effect mobilities of 3×10^{-3}, and 1.5×10^{-2} cm^2/(V·s), respectively, under some conditions. In 1997, Bao et al.[14] discovered that it was possible to achieve high FET performance with phthalocyanine copper with a maximum field-ef-

fect mobility of ca. $0.02 \ cm^2/(V \cdot s)$, and in the following year, he reported a series of researches on metallophthalocyanines[4] (shown in Fig. 1), among which hexadecahalogenated metallophthalocyanine ($F_{16}CuPc$) had a field-effect mobility of $0.03 \ cm^2/(V \cdot s)$. Apparently different from former materials, $F_{16}CuPc$ not only was air-stable but also had the highest field-effect mobility reported at that time, showing a great potential for practical application. Furthermore, Bao found that the field-effect mobility was strongly dependent on metal coordination and the substrate temperature for deposition. X-ray diffraction measurements indicated that $F_{16}CuPc$ molecules in deposited thin films were highly ordered and were standing and essentially perpendicular to the substrate surface. This rendered the π-overlap direction of $F_{16}CuPc$ the same as the current flow direction, which provided an efficient path for charge transport. Recently, it was reported that $F_{16}CuPc$ exhibited a
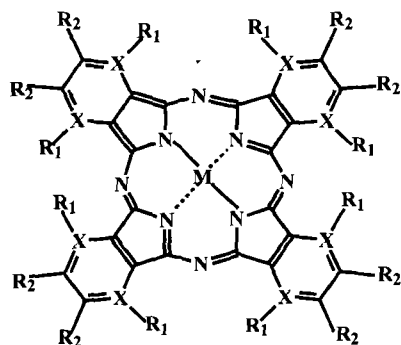
$F_{16}MPc$, $R_1 = R_2 = F$, $X = C$, $M = Cu$, Zn, Fe, Co
$C_{16}FePc$, $R_1 = R_2 = Cl$, $X = C$, $M = Fe$
$(CN)_8CuPc$, $R_1 = H$, $R_2 = CN$, $X = C$, $M = Cu$
$PyCuPc$, $R_1 = R_2 = H$, $X = N$, $M = Cu$

Fig. 1. Molecular structures of phthalocyanines.

mobility of $1.7 \ cm^2/(V \cdot s)$ at room temperature, near to the performance of a-Si:H[15].

1.1.2 Perylene derivatives   It has been a long time since the properties of electron transport in perylene derivatives were studied. Ostrick et al.[16] showed perylenetetracarboxylic dianyhydride (PTC-DA) transported electrons with a mobility of $10^{-4} \ cm^2/(V \cdot s)$. Horowitz et al.[17] investigated the compound of N, N'-diphenyl-3, 4, 9, 10 -perylenetetracarboxylic-diimide (DDP) (shown in Fig. 2, a) and the field-effect transistor fabricated with it. Owing to the instable electron transport property [$\mu_{FET} = 1.6 \times 10^{-5} \ cm^2/(V \cdot s)$], the performance of the device degraded rapidly with time when exposed to air and furthermore the field-effect property completely vanished after two days, which was a common character of many organic materials. The TPPI compound (shown in Fig. 2, d) was investigated by Ranke et al.[18] with the time-of-flight (TOF) technique, and electron mobility of $10^{-5} \ cm^2/(V \cdot s)$ was measured in the bulk. The above dye was also used to fabricate single layer LEDs, showing clearly visible red electroluminescence. Another novel series of perylene derivatives (shown in Fig.2, e~h) were synthesized by Craats et al.[19], which exhibited electron mobilities in the range from 0.01 to $0.1 \ cm^2/(V \cdot s)$, and by varying the alkyl substituents, the structure of the perylene units could be changed from a two-dimensional layered arrangement to one-dimensional. In addition, compounds of DPP-PTCDI[20] (Fig.2, c) and DMe-PTCDI[20] (Fig.2, b) were studied by other researchers, but their charge transport mobilities were not reported.

a $R_1=H$ $R_2=$⬡
b $R_1=H$ $R_2=Me$
c $R_1=H$ $R_2=$⬡
d $R_1=$o-⬡ $R_2=$⬡

e   X=Y=Z=H
f   X=Y=$C_{18}H_{37}$ Z=H
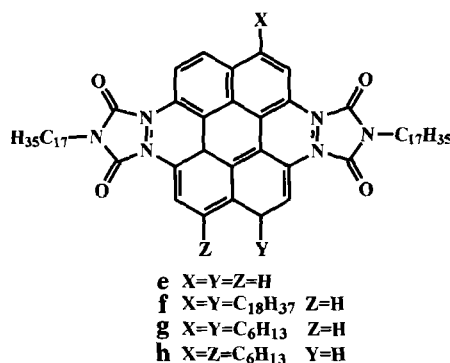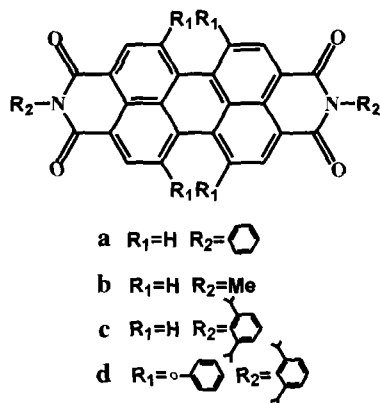g   X=Y=C_6H_{13} Z=H
h   X=Z=C_6H_{13} Y=H

Fig. 2. Molecular structures of perylene derivatives.

1.1.3 Naphthalene derivatives   A new OETM, N-ethyl-4-acetylaminonaphthalimide (EAAN) (Fig.

3, a) was synthesized as the electron transport layer in organic EL devices by Liu et al.[21], which showed

good fluorescence quantum efficency[22], anti-photo-chemical and anti-oxidation properties. Its nice electron mobilities led to an excellent luminescent efficency and brightness in the device.

Fig. 3.   Molecular structures of naphthalene derivatives.

Although 1, 4, 5, 8-naphthalene tetracarboxylic dianhydride (NTCDA) (Fig. 3, b)[23] is much less extensively conjugated compared to the perylene compounds and its crystal structure indicates only partial intermolecular overlap, it has an accessible LUMO for electron injection with an electron mobility up to 3×10^{-3} cm^2/(V·s). The high mobilities were observed under vacuum. However, a 1~2 order of magnitude decrease in electron mobilities was noted in air. Meanwhile different mobilities were also observed because of different material purities and substrate temperatures. NTCDI (Fig. 3, c), having a similar molecular structure with NTCDA, was reported to have electron mobilities up to 10^{-4} cm^2/(V·s), an order of magnitude lower than NTCDA[24]. The other compounds (d, e, f in Fig.3), which also have the naphthalene framework, exhibit soluble and air-stable properties with high electron mobilities[23]. Mobilities for compound d were up to 0.1 cm^2/(V·s) in air, and for e up to 0.01 cm^2/(V·s). Compound f showed no field-effect transistor activity in air, even though its film was highly crystalline. However, degassing it in a turbopumped chamber and testing under high vacuum resulted in a progressively increasing mobility: 0.001 (in 6 h), 0.01 (in 1 day), 0.08 (in 3 days), and 0.16 cm^2/(V·s) (in 4 days).

1.1.4   Polycenes   In the last few years, pentacene was found to be one of the materials with higher charge mobilities. In pentacene single crystal at room temperature, the field-effect mobility of electrons is 1.7 cm^2/(V·s), and 300 cm^2/(V·s) at near absolute zero temperature[5]. Recently Schon[15] showed that its μ_{FET} is 1.9 cm^2/(V·s) at room tem-

perature and 6×10^4 cm^2/(V·s) at low temperature. The electron transport property of tetracene is similar to that of pentacene, with μ_{FET} of 1.6 cm^2/(V·s) at room temperature[15].

1.2   Polymer electron transport materials

Aimed at solving the problems of ease in crystallizing and phase separation, it is an effective way to synthesize polymer electron transport materials with fine thermal stability and processability.

1.2.1   Oligothiophenes   A lot of investigations have been reported on oligothiophenes as the semiconductor layer in thin film transistors[25~28], but most of them essentially behave as p-type semiconductors because electron-richness of the thiophene core facilitates hole injection. By adding fluoride group to thiophene structure, thiophene oligomers can be made as n-type semiconductor, and they become significantly more chemically and thermally stable, such as α, ω-diperfluorohexylsexithiophene (DFH-6T) [μ_{FET} = 0.01 cm^2/(V·s)] (shown in Fig.4) reported by Antonio. In 2001, Sakamoto[6] developed a new OETM: perfluoro-α-sexithiophene (PF-6T) (shown in Fig.4). Its dihedral angles between thiophene rings are very small, which are favorable for π-π overlaps to form electron transport channels. In spite of no report about its mobility, the X-ray structure of PF-6T suggested a high electron mobility along the π-π stacking direction.

DFH-6T

PF-6T

Fig. 4.   Molecular structures of oligothiophenes.

1.2.2   Perfluorinated phenylenes   Recently, π-conjugated perfluorinated phenylenes have received considerable attention as OETMs. In 2000, Sakamoto et al.[29] designed a new perfluorinated phenylene dendrimers (shown in Fig.5) for OLEDs because of (i) their low LUMOs and HOMOs, which are important for electron injection and hole blocking, respectively, (ii) relatively low sublimation tempera-

ture, which makes it possible to deposit high molecular weight compounds with high glass transition temperatures, and (iii) thermal and chemical stability due to strong C-F bonds. Owing to their branched structure, they are expected to favor an amorphous morphology in thin films and to be used as the electron-transport layer for OLEDs. Afterwards Heidenhain et al. [30] reported the synthesis of a series of perfluorinated oligophenylenes, which are better electron transporters than the conventional electron transport materials such as Alq$_3$.
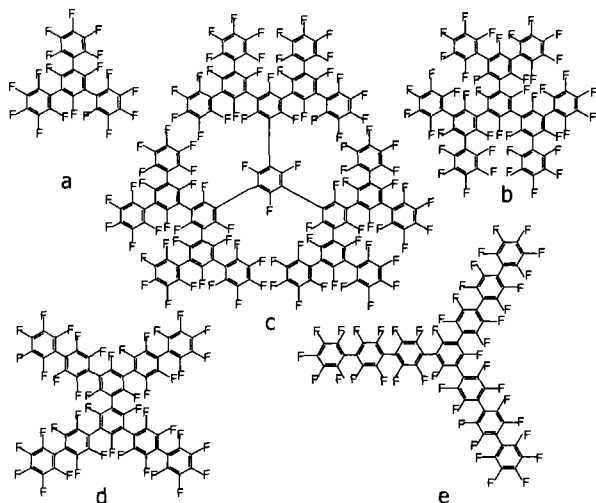
Fig. 5.   Molecular structures of perfluorinated phenylenes.

1.2.3 PPV derivatives   PPV is a class of excellent material for OLEDs, however, its ability to transport holes is better than that to transport electrons, which minishes the luminescent efficiency greatly. Weaver[31] introduced electron-drawing groups or electron deficient nitrogen atoms into the structure of PPV, such as CN-PPV and PpyV-co-PDHepOPV, so as to evidently improve the electron transport mobility and luminescent efficiency. In 2001, a kind of novel PPV derivative containing electron-transporting group on the main chain (O-PPV) was synthesized by Yang et al. [32], and a single layer light-emitting device was fabricated. Compared with the quantum efficiency of PPV, it is about eight times higher, which can be associated with the balanced carrier injection and transport owing to the attachment of oxadiazole segment onto PPV main-chain.

1.3 Others

The fast development of polymer electroluminescence devices promotes the development of electron

transport polymers. Zhang et al. [33] showed a compound of poly (phenyl quinoxaline) (PPQ) (Fig. 6) used in the organic polymer heterostructure LEDs, which enhanced the brightness of device by nearly two orders. PPQ was identified as a potential electron transport material due·to the presence of quinoxaline moieties containing electron deficient nitrogen in the polymer chains. This conjugated electronic structure provides high environmental chemical and thermal stability. The function of compound of 1, 3-bis[5-(p-t-butylbenzyl)-1, 3, 4-oxadiazolyl-2] benzene (PDPDP-Bu-t) in the OLEDs was investigated by Hamada et al. [34], which exhibited an improvement of quantum efficiency by balancing the nearly equal injections of holes and electrons. In recent work, Cea[35] reported on two oxadiazole derivatives PDPy containing a pyridine unit as new OETMs applied in single layer OLEDs. The investigation of effect of blending these materials with MEH-PPV in devices showed an external quantum efficiency enhancement compared to the ITO/MEH-PPV/Al structure, which could be explained by the fact that a hybrid of pyridine and oxadiazole led to increased electron-injection ability.

Some work[24] on 11, 11, 12, 12-tetracyanonaphtho-2, 6-quinodimethane (TCNNQ) (Fig. 6) has indicated that its electron mobilities are higher than tetracyanoquinodimethane (TCNQ) ($\mu_{FET} = 3 \times 10^{-5}$ cm^2/V·s) with a greater stability in air. In 2001, a non-dispersive and air-stable electron transport semiconductor based on silole derivatives PyPySPyPy (Fig. 6) was reported by Murata[36], who found that electron mobilities of PyPySPyPy was a function of the square root of the bias field, its mobility ranged from 9×10^{-5} to 2×10^{-4} cm^2/V·s when E$^{1/2}$ was in the range from 500 to 800 (V/cm)$^{1/2}$. Perepichka et al. [37] synthesized a novel push-pull dithiole-fluorene compound DTNF as electron transport material for holography. The solubility problem was solved by introduction of a long-chain alkoxycarbonyl substituent into the fluorene ring and a comparative investigation of photoconductivity sensitization of DTNF showed that intramolecular charge transfer in the acceptor led to a significant increase in the electrophotographic response. Moreover, PdPhQx[38], BBOT[39], dibenzopuinone, and benzofluorene were also reported as organic electron transport materials.
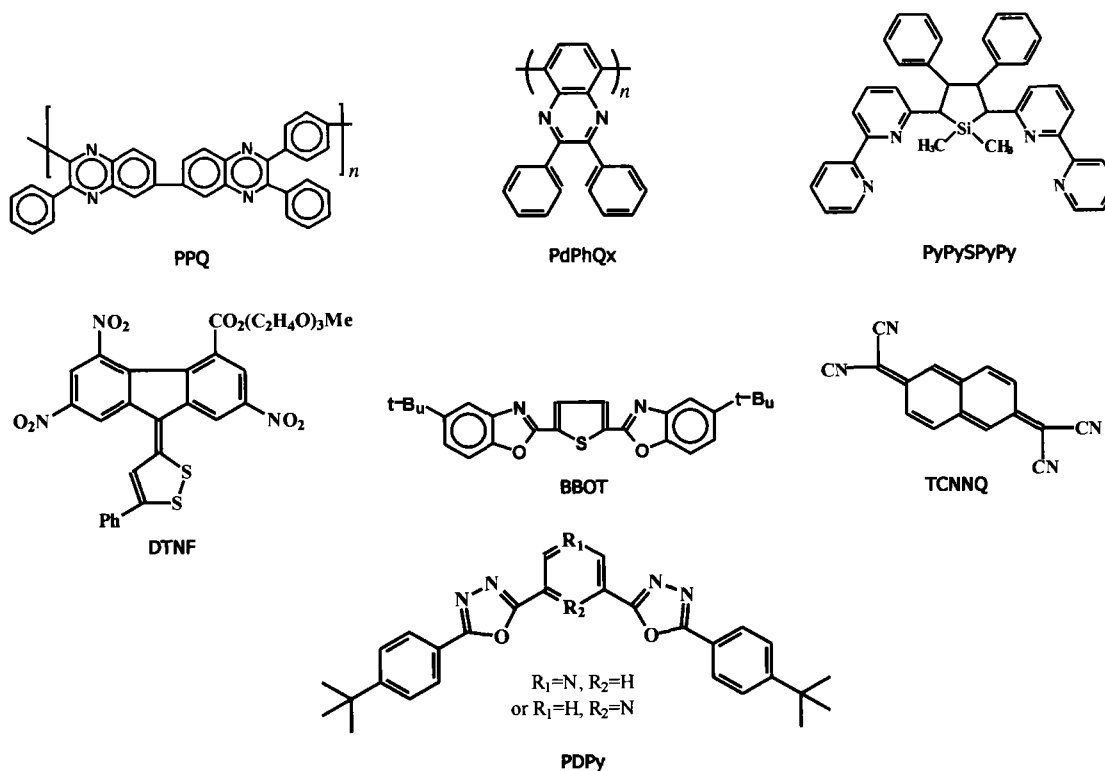
Fig. 6. Molecular structures of other compounds.

2 Measurement technologies for charge carrier mobility of OCTMs

Carrier mobility is one of the most important parameters to characterize OCTMs. But because of small mobilities, most of the usual methods applied to inorganic semiconductors are not suitable to organic semiconductors. Up to date, there are four technologies used to measure carrier mobility in organic materials: charge dissipation method[40], transient current method[41], time-of-flight (TOF) method[42], and field-effect method[24]. The drifting mobility (μ_d) can be obtained by the former three technologies, while field-effect mobility (μ_{FET}) obtained by the fourth. Presently the last two technologies are most often used.

In the technology of charge dissipation method[40], thin film is charged by short exposure to a negative corona in air and the decay of the field of charges is followed by a fieldmeter. The fieldmeter is calibrated in terms of potential with reference to earth and the immediate potential of the experiment is curved against time. It was found that the charges deposited on the film provided not only the driving field for the conduction process, but also a large proportion of the transported charges. The transient current method[41] assumed that carriers drifting decayed primarily by recombination. The specimen films with both surfaces metallished, were bombarded through one of the electrodes. Then the transient current is recorded and the carriers distribution is obtained. TOF[42] is a traditional technology of measurement, also a popular one, by which photoinduced quantum efficiency, charge carrier mobility and other information during the charge transport procedure can be obtained. It can be applied not only to single crystals and polycrystals, but also to polymers. In this method, film sample is charged under an electric field and excited by a light pulse which created pairs of charge carriers near the electrode, then the electric field due to different polarity transports only one of the carrier components to the counter, leaving the other. During the transport procedure, the current signal is recorded by circuitry. The above three technologies of measurement are summarized in Table 1.

Table 1.   Technologies used to measure the carriers drifting mobility in organic materials

	Charge dissipation method	Transient current method	TOF method
Characteristic	Depending on the rate of decay of surface potentials	Analyzing transient current excited nonuniformly in surface layer	Measuring the drifting time of superfluous carriers across sample film
Applicable condition	Decay of surface potentials being originated from exoteric injected charge and ignoring carrier traps	Based on the theory of carriers doping model; ignoring deep traps and carriers drifting decayed primarily by recombination	Time of carriers formation $\ll t_T$, thickness of carriers formation layer $\ll L$, uniformly distributed electric field, $RC \gg t_T$, ohm contacts with electrode
Disadvantage	Residual potential and carrier traps leading to inaccurate testing results	Being difficult to confirm the penetration depth (b) of carriers	Being difficult to prepare samples and electrode and testing apparatus is expensive
Advantage	Easy operation	Feasible to ignore space charge effect	Accurate testing results and more information obtained
Calculating formula	$dV/dt = -\dfrac{1}{2}\mu_d(V_0/L)^2 = -\dfrac{1}{2}\mu_d E_0^2$	$\mu_d = bL/Vt_t$	$\mu_d = L/t_T E$

Note: E_0 is initial electric field intensity, E is electric field intensity, V_0 is initial potential, V is bias voltage, L is film thickness, t_T is time of flight.

μ_{FET} can be calculated from the parameters received in the testing of field-effect method[24] with the following equation:

$$I_{DS} = \frac{WC_i}{2L}\mu_{FET}(V_G - V_0)^2 \qquad (1)$$

where L and W are the channel length and width of field-effect transistor, respectively, C_i is the capacitance per unit area of the oxide, V_0 is the extrapolated threshold voltage, V_G is the gate voltage, I_{DS} is the drain-source current, and μ_{FET} is field-effect mobility. Compared to TOF, the field-effect method has its own peculiarity. Because μ_{FET} is obtained in testing film field-effect transistor device, it closely depends on the field intensity, gate voltage, and processing technics of FETs besides the compound's own mobility, whereas, μ_d is the intrinsic mobility of compound. We can judge that charge carriers are electrons or holes by the current-voltage characteristics of the field-effect transistor: n-type semiconductor when positive voltage in the gate, otherwise p-type when negative voltage.

3   Design of organic electron transport materials

It is known that for a material to transport electrons (n-type), it needs to have an accessible LUMO level for electron injection and sufficient π-overlaps to achieve reasonable charge carrier mobilities. Therefore, molecules with strong electron-withdrawing groups and extended π-systems are good candidates as n-channel semiconductors[4].

Generally we can adopt two design criteria for electron transport molecules, namely (i) incorporation of strong electron-drawing groups or electron deficient nitrogen atoms and (ii) construction of non-dipolar structures. The rationale for the former is that the principal problem with regard to poor electron transport is the presence of electron traps: adsorbed oxygen is one likely culprit. Thus, one approach to improving electron transport is to synthesize materials having a high electron affinity so that the electrons become more difficult to trap. Non-dipolar structures are preferred to avoid dipole scattering of carriers which would tend to reduce their mobilities: this is a well-known design rule for electronphotographic charge transport materials.

Electron transport of OETMs in thin films depents on a number of factors such as molecular orientation, electronic overlap, molecular orbital energy levels, traps, and sometimes the nature of the ambient. For example, thin films of PTCDA[16] possess charge transport anisotropies in the species of charge that can be transported in different directions. It is found that PTCDA transport electrons in the directions parallel to the molecular planes and mainly holes in the directions perpendicular to the molecular planes. Therefore, besides molecular structures, the aggregate structure of materials is also a key factor to the design of high performance OETMs.

In addition, n-type materials can be obtained by doping or modification of p-type ones. Presently it is a hotspot of research to convert a p-type organic semiconductor to an n-type one, which indicates an effective way to obtain OETMs.

4 Conclusion

The organic electron transport materials mentioned above are classified into a couple of kinds, their recent trends and developments are summarized, and the measurement technologies for their charge carrier mobility are introduced. It is found that the charge carrier mobilities of OETMs are potential to be improved; OETMs are not air-stable, namely an apparent decline of their performance when exposed to air; the material design principles are suggested for synthesizing and developing a new class of OETM; TOF and field-effect methods are the most popular ones for characterizing charge carrier mobility of organic materials presently.

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