PERFECT TONERS HIDE A SECRET:

HDK® – Pyrogenic Silica
HDK® PYROGENIC SILICA: TOTAL CONTROL OVER TONER CHARGE AND FLOW
Your expectations of an electrophotographic toner are highly specific – HDK® is no less specific in influencing your toner. This pyrogenic silica tunes tribocharging properties and flow selectively, efficiently and reliably. And that is the HDK® secret to optimized toners.

Unique product properties
WACKER has been producing HDK® pyrogenic silica for more than 40 years and, for 30 of those, it has produced dedicated grades for toner use. And we continue to specifically enhance the unique properties of the HDK® range.

The pay-off: HDK® effectively and selectively optimizes the charge and flow properties of a wide variety of toners, whether made by classic melt/grinding or by new chemical methods.

The electrophotographic process is based on two natural phenomena: materials of opposite charge attract, and certain materials become more electrically – conducting when exposed to light.

Principle behind the electrophotographic process

\[ F = Q \times E \]

where:
- \( F \) = force
- \( Q \) = charge
- \( E \) = field strength
**Toner Production**

Generally, a crude toner consists of a binder resin, a colorant and additional technical ingredients (CCAs, waxes). This crude toner is then coated with a small quantity of flow improver (HDK®) in the additive blending process. There are basically two different ways for making the crude toner:

**Melt-grind**

The main steps are melt mixing, grinding/jetting and classifying. This method makes toner by pulverizing chunks of toner compounds and sieving them to collect the proper size of toner particles. This process leads to irregularly shaped toner particles with mean sizes between 6 to 12 micrometers. The end product is a “conventional” toner.

**Chemical**

This process follows the path from molecule to solid particle, with the particles growing under controlled conditions. These may be made either by emulsion polymerization of resin monomers or by controlled growth of latex particles. This process leads to regularly shaped toner particles (spheres, “potatoes,” etc.) with mean sizes between 5 to 7 micrometers. The end product is a “chemical” toner.

To satisfy the highest quality demands of this market and to accommodate the rising diversity of modern toners, we have developed a unique product matrix. This matrix enables HDK® grades to be selected according to stringent key parameters:
- HDK® – particle size
- HDK® – surface chemistry
- HDK® – tribocharging

**Concentrated expertise**

An innovative, highly motivated partner to the non-impact printing industry, we not only supply products of the utmost quality, but also stay abreast of technological development through our own scientific contributions. We have obtained patent protection for our products and their use in the field of non-impact printing (NIP). For more than a decade, we have participated actively in international NIP platforms, such as IS&T, Japan Hardcopy and IMI.

We are also collaborating closely with Clariant, a major international player in the field of toner pigments and charge-control agents, to boost our global market presence. With the Clariant partnership, we are positioned as an expert supplier of an impressive portfolio of pigments, charge-control agents and HDK® pyrogenic silica.

Moreover, we provide thoroughly reliable, personalized advice worldwide and a full package of technical support on location.
HDK® enables toner flow and charge properties to be formulated especially efficiently and economically. We have created a comprehensive, dedicated range of HDK® grades that are extremely effective at the smallest of loadings. An overview of this range is presented on pages 16 – 18.

HDK® particle sizes
Particle size is inextricably linked to the unique process by which the pyrogenic silica is made.

The aggregates are formed by the collision of primary particles during particle growth. The aggregates represent the smallest, most stable, non-dispersible particle unit of three-dimensional structure. Nevertheless, the freely – available BET surface area ensures that the silica adheres extensively on the toner particles, and that even applies to the aggregates.

HIGH PERFORMANCE NEEDS A STRONG FOUNDATION: HDK® PYROGENIC SILICA

Scanning electron micrograph (SEM) of a CPT particles covered with HDK® particles

HDK® pyrogenic silica – the production process

Particle formation of HDK® pyrogenic silica

<table>
<thead>
<tr>
<th>Type</th>
<th>Size (nm/µm)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primary particles</td>
<td>5 ............ 50</td>
<td>Influence on BET surface area</td>
</tr>
<tr>
<td>Aggregates</td>
<td>0.1 ........... 0.5</td>
<td>Actual particle size on the toner surface</td>
</tr>
<tr>
<td>Agglomerates</td>
<td>1 ............. 250</td>
<td>A narrow distribution ensures even reduction in the mixing process to the final aggregate size.</td>
</tr>
</tbody>
</table>

Note: Definitions of primary particles, aggregates and agglomerates are those contained in DIN 53206 page 1 (08/72).
PARTICLE SIZE

WACKER deploys the latest manufacturing technologies and integrated production workflows. These safeguard HDK®’s extraordinary versatility and guarantee that quality remains consistently high.

Targeted particle size control
Under common conditions of technical handling (filling, shipping, storage etc.) the agglomerate is the relevant particle size. During the critical additive-blending process for making toner, these are reduced to the size of aggregates. This ensures optimum coverage of the toner surface with HDK® particles.

It is highly advantageous to the additive-blending process to have a narrow agglomerate size distribution and for agglomerates to be as small as possible. To this end, a grinding-classifying stage is incorporated into the process line.

The advantages:
- Easy dispersing
- Shorter mixing times
- Homogeneous distribution of HDK® aggregates on the toner surface
- Fast, stable charging of the toner

<table>
<thead>
<tr>
<th>Agglomerate size distributions, volume density distribution (q3) and volume cumulative distribution (Q3) of toner HDK® vs. a commercial standard silica for toner use.</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Graph showing particle size distributions" /></td>
</tr>
</tbody>
</table>

Process control station: HDK® for toner use is produced on our own specially-developed process lines.
HYDROPHOBIZATION AND FUNCTIONALIZATION

Special surface treatment
HDK® with an unmodified surface has 2 silanol groups per square nanometer (nm²) and so is hydrophilic. This leads to high surface energy and high levels of water absorption.

Toners must continue to possess free flow and a stable charge at high atmospheric humidity and elevated temperatures. That is where hydrophobic HDK® comes in, with its much reduced density of silanol groups (<0.5 per nm²). Its surface energy is significantly lower and it absorbs noticeably less moisture.

As leading producer of silanes, silicones and HDK® pyrogenic silica, WACKER deploys the latest knowledge from organosilicon research to optimize and further advance the surface chemistry of HDK®.

Our development goals are:
• Chemical immobilization of the organosilicon component on the HDK® surface
• Even distribution of the organosilicon component over the HDK® surface

The surface energy of hydrophobic HDK® can be determined by Stevens’ method, which measures the miscibility of a powder in water/alcohol mixtures. The variable here is the volume of alcohol needed for wetting the powder. A high alcohol consumption means that the solid has a low surface energy.

The methanol wetting test can be used to grade the levels of hydrophobization. Functionalization of the HDK® surface with certain chemical groups not only is useful for imparting hydrophobic properties, but also affords a way of obtaining the desired level of tribocharging.

The HDK® product portfolio for toner use comprises:
• Surface-modified HDK® ranging from low specific BET surface area (large particles) to a high specific BET surface area (small particles)
• HDK® surface-treated with dimethyl dichlorosilane, hexamethyldisilazane, PDMS for negative tribocharging
• HDK® functionalized with amino/ammonium end groups for positive tribocharging
• Development of customized products for specialty requirements (minimum order quantity required)

Using HDK® to formulate toners is highly economical because it is so efficient and processing is simple. The special mechanical de-agglomeration pretreatment step makes the HDK® easy to disperse so that it distributes itself evenly over the entire toner surface. Excellent results are obtained at minimum loading with 0.2 to 2 %.

The benefits:
• Significant improvement in toner flow
• Better toner transfer from the developer unit to the organic photoconductor (OPC)
• Precise control over tribocharge
• High charge stability
• Selective functionalization, i.e., high positive or negative charge on the highly hydrophobic HDK® over all BET surface areas
• Easier cleaning of the OPC
• Better transfer of toner to the substrate
A critical parameter is how strongly the HDK® particles adhere on the toner surface. If the adhesive forces are too weak, the HDK® will be lost. Furthermore, the toner loses its ability to flow and its electrostatic charge also changes.

In the end, print quality suffers. We work closely with external research institutions (e.g., Ulm University) to quantify the adhesive forces involved. Our research involves a scanning force microscope.

Scanning force microscopes use a tiny cantilever to scan the sample – here, a toner particle – line by line. Aside from revealing the topography, contact between the tip and the sample’s surface enables additional properties, such as hardness and adhesion, to be determined due to a phenomenon known as phase displacement.

Scanning force micrograph (SFM) in phase displacement of a conventionally – produced polyester toner containing 0.5 wt% HDK® H30TD

**Phase displacement**

“Harder” HDK® particles show up clearly in phase-displacement images as “bright” particles. A loading of 0.5 wt% of a 50 m²/g HDK® is not enough to completely envelop the toner’s surface. But a 300 m²/g HDK® (HDK® H30TD) at 0.5 wt% produces almost total coverage.

References:
REGISTRATION AND TOXICOLOGY

HDK® pyrogenic silica registration
All HDK® grades for toner application have been chemically – registered with the relevant national and international authorities.

HDK® pyrogenic silica toxicology and safety
Like all synthetic, amorphous silicas, HDK® is classified as non-toxic when used as intended. Details are provided in the relevant safety data sheets.
EFFICIENCY IS THE RESULT OF EXACT MEASUREMENT

Intensive, fundamental scientific research by WACKER is used to continually develop specialty HDK® grades of very narrow particle size distribution (agglomerates), different tribocharge and higher flow that make your products successful in your market.

Flow properties
To measure the flow performance of HDK® or toner/HDK® systems we are using a commercial Shear Scan Powder Flowability Analyzer which enables us to quantify cohesion, flow index values, angle of internal friction and other flow characteristics.

HDK® H30TD has less strength than HDK® H05TD under all loads (kPa). HDK® H30TD flows better. Toner/HDK® system based on BET 300 m²/g will flow the best.

Toner HDK® fundamentally improves toner flow. The cohesion forces between the toner particles are significantly reduced by the “surface layer” HDK®.

The best tool for comparing powder behavior is the flow function. This is a plot of a material’s strength versus consolidation stress. The powder with the lowest strength at any given stress will flow the best.
Charge spectrometers

q/m-mono device; Epping GmbH; Germany
In use, each toner/HDK® system is subject to extensive shearing, sometimes under critical environmental conditions. We estimate the flow of such “aged” toner/HDK® systems using a special charge/flow tester (q/m-mono device; Epping GmbH; Germany).

The toner collected inside the cell (isolated faraday cage) is used not only for determining the charge, but also for measuring the flow of the toner/HDK® system. Prior to the measurement, the toner system is aged by moving it past a doctor blade for a defined period, equivalent to several thousand photocopies. The more toner that ends up in the measuring cell, the better the flow.

By using the same amount of HDK®, the flow of the toner improves with increasing BET surface of the corresponding HDK®.

References:
Charge properties

The intrinsic charge of the silica is measured by blowing silica off iron carrier particles. Most HDK® toner grades have a high negative tribocharge. HDK® grades with a high BET surface area undergo the most charging. The HDK® surface charge can be changed to positive by functionalizing the HDK® with amino/ammonium groups.

We use a Q-Test charge spectrometer provided by Epping, Germany, to conduct detailed studies.

In addition to the charge mean value, it is possible to measure the charge of each individual toner particle.
For example, in a negative toner system opposite charged toner particles may lead to inferior photocopy images. Their proportion in the toner can be reduced through judicious choice of HDK®.

Consistent with accepted theory, small HDK® particles (larger BET surface area) prove to be more effective at charging the toner/HDK® system.

In all cases, a full charge is quickly attained and it remains stable for the duration of the study.
The surface chemistry of HDK® influences the system’s charge (positive or negative), the level and its stability. Silica/toner systems are renowned for their unstable charges, especially at high atmospheric humidity and elevated temperatures. Assuming that the toner and silica are blended under optimum conditions, the cause of the instability may be related to poor or uneven hydrophobization of the silica. This is why WACKER focuses on optimized processes for hydrophobizing HDK®.

**HDK® surface chemistry vs. toner charge**

![Graph showing the relationship between HDK® surface chemistry and toner charge](image)

**Triboelectric charge stability of toner/HDK® systems at low/high atmospheric humidity and low/elevated temperature and increasing stress time – 0.8 wt% HDK®**

![Bar chart showing triboelectric charge stability](image)

With HDK®, a higher charge level of the toner is achieved; the highest level with the toner/HDK® development combination. In the standard HDK®/toner system a slight decrease of the charge over time occurs. The toner/HDK® development system shows a nearly stable charge over time even under H/H conditions.
The comprehensive HDK® range satisfies all charge stability and flow requirements. The following tables provide an overview of the offerings. Please contact WACKER for detailed information.

**THE RIGHT HDK® PYROGENIC SILICA GRADE FOR EVERY TONER**

HDK® with negative tribocharge

<table>
<thead>
<tr>
<th>HDK®</th>
<th>H1303VP</th>
<th>H2000/4</th>
<th>H2000T</th>
<th>H3004</th>
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1) DIN ISO 9277; DIN66131; Hydrophobic BET surface areas do not lend themselves to determinations of particle size
2) DIN EN ISO 787-9; 4% in methanol / water 50:50
3) DIN EN ISO 787-11
4) Primary particles of silica do not occur as individual units
5) Laser diffraction; easily dispersed to submicron sized aggregates in the additive blending step
6) Relative silanol content with respect to hydrophilic pyrogenic silica (containing 2 SiOH/nm²)
7) Blow off vs. ferrite; WACKER method

Note: These figures are intended as a guide and should not be used in preparing specifications.
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<th>H30TD</th>
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<td>200 ± 30</td>
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<td>4 – 7</td>
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<td>&lt;20</td>
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<td>5 – 8</td>
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<td>5 – 8</td>
<td>5 – 8</td>
<td>5 – 8</td>
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<td>[g/l]</td>
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<td>approx. 70</td>
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<td>[µm]</td>
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</table>

1) DIN ISO 9277; DIN66131
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7) Blow off vs. ferrite; WACKER method

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HDK® with positive tribocharge

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<tr>
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</table>

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Abbreviations:
- HMDS = Hexamethyldisilazane Si–O–Si(CH₃)₃
- DMDS = Dimethylidichlorosilane Si–O–[Si(CH₃)₂Cl]–O–¹⁺
- PDMS = Polydimethylsiloxane Si–O–[Si(CH₃)₂O]–¹⁺

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Note: These figures are intended as a guide and should not be used in preparing specifications.
Compelling performance
This brochure shows you what HDK® can do for toners. Obtainable on request are detailed product leaflets expanding on the overview information of pages 16/17/18. Alternatively, drop by our internet site at www.wacker.com/HDK. We use technology platforms such as IS&T and Japan Hardcopy to present our innovations.

The NIP industry is a strategic target market for WACKER. Intensified research and development and a growing range of new products are evidence of our commitment.

Local customer service
Our local experts see to it that your innovations are conducted by our own product developments. Our technical core team coordinates all activities and provides assistance ranging from classical technical support to fundamental research through to supply chain support.

If you are seeking solutions for conventional black or color toners or for chemical toners in any color category, our HDK® range offers the right grade for all your needs – hydrophobic or functionalized, and customized to your requirements. Our specialists will gladly assist you with choosing the right HDK® grade. They will also tell you more about the technical support we offer.

Our sales partners Clariant Japan K.K. in Japan and Clariant Corporation in the NAFTA are always on hand as well to provide further information (www.nip.clariant.com).

Our service program
• Personalized advice
• Custom development of modified HDK®
• Comprehensive analytical characterization of the silicas
• Determination of the hydrophobic level of modified silicas (methanol test).
• Determination of the triboelectric properties of silicas and toner/silica systems
• Determination of the morphology of toner/silica systems
• Determination of the flow properties of toner/silica systems
• Global chemical registration of HDK®
• Use of the latest technology in the production of HDK® for ensuring safe, ecologically sound manufacture
• We are committed to the principle of sustainable development

Global Presence
The NIP industry is a global one. And that is how we have aligned ourselves. One face to the customer.
WACKER is one of the world’s leading and most research-intensive chemical companies, with total sales of €4.63 billion. Products range from silicones, binders and polymer additives for diverse industrial sectors to bio-engineered pharmaceutical actives and hyperpure silicon for semiconductor and solar applications. As a technology leader focusing on sustainability, WACKER promotes products and ideas that offer a high value-added potential to ensure that current and future generations enjoy a better quality of life based on energy efficiency and protection of the climate and environment. Spanning the globe with five business divisions, operating 24 production sites, WACKER is currently active in over 100 countries. The Group maintains subsidiaries and sales offices in 29 countries across Europe, the Americas and Asia – including a solidly established presence in China. With a workforce of 16,300, WACKER sees itself as a reliable innovation partner that develops trailblazing solutions for, and in collaboration with, its customers. WACKER also helps them boost their own success. Our technical centers employ local specialists who assist customers worldwide in the development of products tailored to regional demands, supporting them during every stage of their complex production processes, if required. WACKER e-solutions are online services provided via our customer portal and as integrated process solutions. Our customers and business partners thus benefit from comprehensive information and reliable service to enable projects and orders to be handled fast, reliably and highly efficiently. Visit us anywhere, anytime around the world at: www.wacker.com

All figures are based on fiscal 2012.
The data presented in this brochure are in accordance with the present state of our knowledge but do not absolve the user from carefully checking all supplies immediately on receipt. We reserve the right to alter product constants within the scope of technical progress or new developments. The recommendations made in this brochure should be checked by preliminary trials because of conditions during processing over which we have no control, especially where other companies’ raw materials are also being used. The information provided by us does not absolve the user from the obligation of investigating the possibility of infringement of third parties’ rights and, if necessary, clarifying the position. Recommendations for use do not constitute a warranty, either express or implied, of the fitness or suitability of the product for a particular purpose.