Use of a Closed Vial Milling System in Investigations of Carbon Black Ink Formulations: it's Advantages and Disadvantages

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Introduction to the problem

Carbon Black in Ink

What is carbon black?

Carbon black is a material produced by the incomplete combustion of heavy petroleum products and is a form of paracrystalline carbon that has a high surface-area-to-volume ratio.

Used as a printing ink pigment (surface area 30 - 138 m^2/g)

Higher BET surface area carbon black also exist

How is the ink made conventionally?

Mixture of carbon black, dispersants, water.

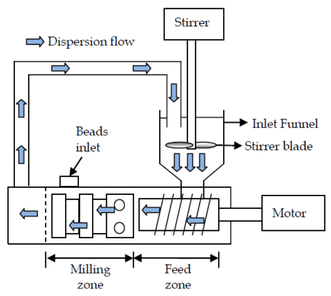
These ingredients are dispersed together under impact and shear in a mill, usually by means of energetic beads (bead mill).

Ink chemistry by Chemistry World1 March 2003
https://www.chemistryworld.com/news/ink-chemistry/3002158.article

A Conventional laboratory process

Lab scale re-circulating bead mill (here a Eiger Torrance Mini motormill 50).

- Sample volume
  - 70 mL (*minimum*)
- Conditions
  - 3750 RPM
  - 3 hours (determined by earlier experiments)



The problem with conventional lab techniques

Number of Ingredients x Number of Levels (concentrations) x nConditions (time, temperature, etc) = Many experiments

Many experiments:

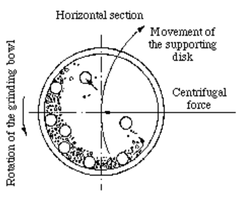
Consumes a lot of ingredients
Produces a lot of waste
Takes a long time
A lot of cleaning

A milling system was sought that could:

Process many samples at the same time
Use smaller samples, but sufficient for testing (~10 ml)
Suitably disperse the sample!

Automaxion Mills

Based on Planetary Bead Milling



Milling jars replaced with vial holder

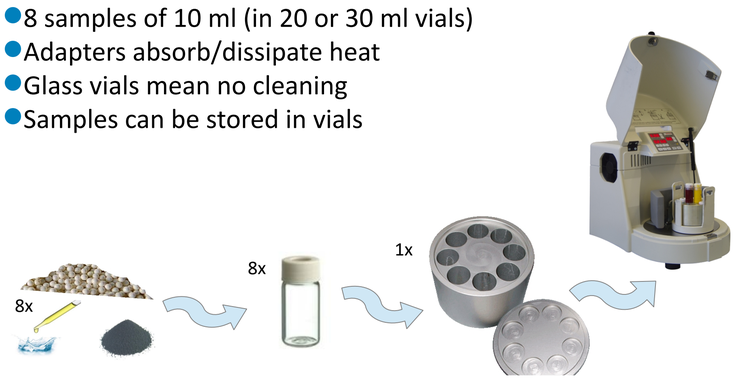
Mill for 10 ml in 'EPA' vials samples selectec

Mills using 1, 2 or 4 adapters available

Experimental

Automaxion single position P6 mill selected

- 8 samples of 10 ml (in 20 or 30 ml vials)
- Adapters absorb/dissipate heat
- Glass vials mean no cleaning
- Samples can be stored in vials



Dispersing additives

High molecular weight non-ionic copolymeric dispersants

Dispersant	Supplier
Solsperse 44000	Lubrizol Corp
BYK-190	BYK-Chemie
Tego 760W	Evonik Tego

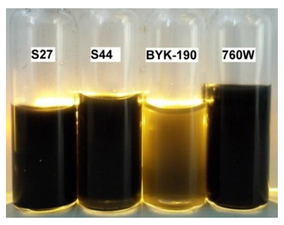
These were selected as candidate materials by quick 'add and settle' screeening experiments

Screening experiments

Sinking test to determine the affinity of dispersant towards pigment

Pigment added to dispersant solution and left for 24 hours without agitation.

The opacity and volume of sediment were qualitatively analysed



Procedure to prepare pigment concentrates

- Step 1: Determine the theoretical amount of dispersant
- Step 2: Determine the maximum pigment loading
- Step 3: Optimise the dispersant amount
- Step 4: Analyse the dispersions

Example results for dispersion of Vulcan XC605 (mentioned as VXC605) carbon black pigment using Tego 760W (760W) as dispersant are presented here.

Analytical techniques

Particle size analysis. Indicates in combination:

if agglomerates are broken up (milling)
if sufficient dispersant is provided (titration)
if dispersant is effective (chemistry)

Rheology: A critical parameter for printing. Indicates:

interactions between the particles, fluids and surfaces, e.g. charge, reversible building of structure, etc.

Storage stability (change in rheology over time). Indicates:

Non-reversible build-up of structure (re-agglomeration)
Migration of ingredients

Instruments

Malvern Zetasizer

TA instruments Rheometer

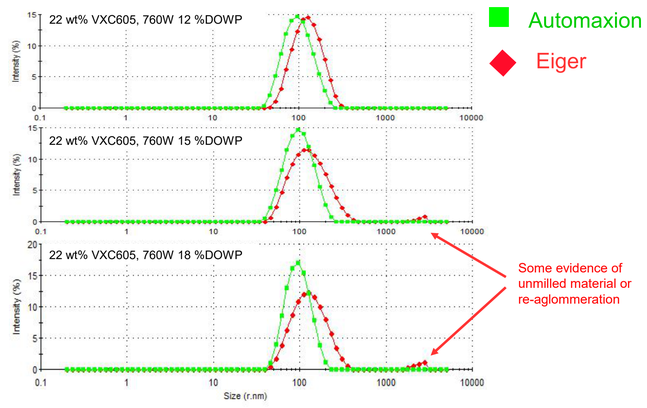
Results

Results (Particle size analysis)

Avg. particle size (nm)		
Dispersion name	Eiger mill	Automaxion mill
VXC605 22 wt%, 760W 12% DOWP	113.8	90.47
VXC605 22 wt%, 760W 15% DOWP	119.3	90.26
VXC605 22 wt%, 760W 18% DOWP	123.3	90.23

DOWP: *D*ispersant *O*n the *W*eight of *P*igment

Results (Particle size analysis)

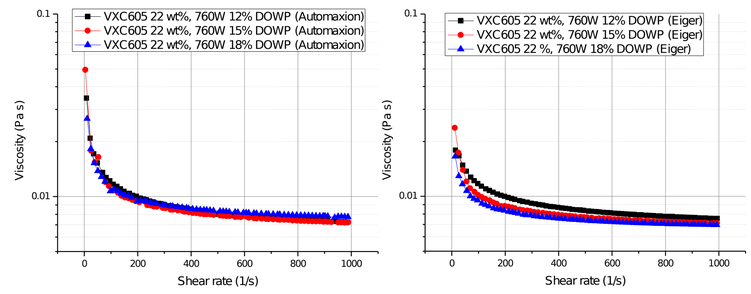


Results (Zeta potential)

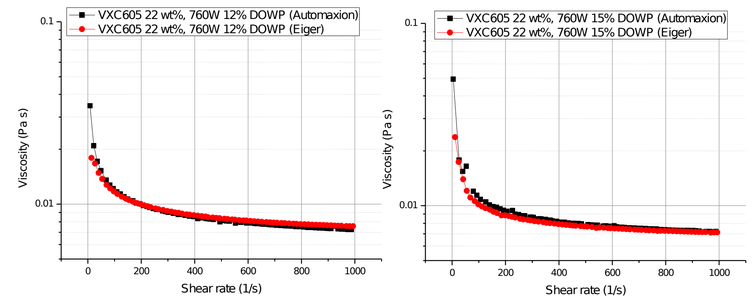
Zeta potential (mV)		
Dispersion name	Eiger mill	Automaxion mill
VXC605 22 wt%, 760W 12% DOWP	-23.2	-17
VXC605 22 wt%, 760W 15% DOWP	-22.3	-29.5
VXC605 22 wt%, 760W 18% DOWP	-21	-33.4

Rheological characterisation

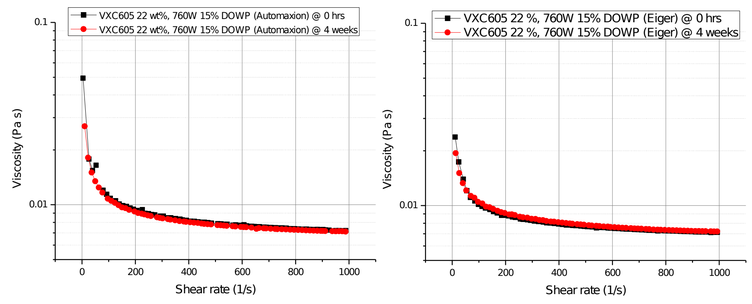
Percentage dispersant effect with different mills



Comparison of mills for two dispersant levels



Storage stability for the two mills



Preparation times (approx.)

Eiger: 15 minutes weighing ingredients and preparation

30 minutes pre-mixing

3 hours milling

30 minutes cleaning

Total = 4 hrs 15 mins *per sample*

3 samples = 12 hrs 45 minutes (or 34 hrs for 8 samples)

Automaxion: 15 minutes weighing ingredients and prep

0 minutes pre-mixing (not done)

4 hrs 30 mins milling for up to 8 samples at a time

<2 minutes bead separation

0 minutes cleaning (not needed)

Total = 4 hrs 47 mins for only 1 sample

3 samples = 5 hrs 20 minutes (or 6 hrs 45 min for 8 samples)

Preparation times (*reality*)

Eiger

1 samples = 4 hrs 15 mins (or 34 hrs for 8 samples)

Reality = 1 sample per 8 hr day

Automaxion (P6)

8 samples = 6 hrs 45 min

Reality = 8 samples per 8 hr day

Or **Automaxion (P5)**

32 samples in day

Conclusions

- The mill is able to make pigment dispersions for printable inks
- Enabled rapid experiments
- Easy to use
- Produced representative inks
- Reproducible
- Some trends in particle size and zeta potential need further investigation
- Unable to process highly viscous samples but they were also difficult using a recirculating mill

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