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Ali, M, Lin, L orcid.org/0000-0001-9123-5208, Faisal, S et al. (2 more authors) (2019) Let-down stability and screen printability of inks prepared using non-printing ink grades of carbon black pigment. Pigment and Resin Technology, 48 (6). pp. 523-532. ISSN 0369-9420

https://doi.org/10.1108/PRT-06-2019-0050

Let-down stability and screen printability of inks prepared using non-printing ink grades of carbon black pigment

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Abstract

Purpose - To analyse the let-down stability of the binder-free dispersion of non-printing ink grades of carbon black and to assess the screen-printability of the finished inks formulated thereof from these pigment dispersions.

Design/Methodology/Approach - Binder-free pigment dispersions that were prepared and optimised following a ladder series of experiments (reported in a separate study by the authors) were let-down with three different binders such that inks containing various amounts of a binder were prepared followed by a rheological characterisation immediately after formulation and after four weeks of storage. The screen printability of the inks that displayed considerable stability was assessed, so was the ink film integrity.

Findings – The pigment dispersions that were considered in the present study were generally found to be stable after let-down with different binders. This was indicated by the fact that the finished inks possessed a shear thinning viscosity profiles, after formulation and after storage, in most of the cases. Furthermore, the screen printability of the inks was also found to be good in terms of registration quality of a selected design. The structure of the ink film deposits on uncoated and binder-coated textile fabrics was also highly integrated and free from discontinuities.

Originality/Value – Carbon blacks with very low volatile matter content and/or high surface area are generally not considered suitable for use in the formulation of printing inks. This is because of their generally poor dispersability and inability to form dispersions that remain stable over extended time periods. This work, which is a part of a larger study by the authors, concerns with the stability of inks formulated from binder-free dispersions of such non-printing ink grades of carbon black. The major advantage of using such pigments in inks is that the required functionality is achieved at considerably low pigment loadings.

Keywords

Carbon black, textile printing inks, screen printing, pigment dispersions, viscosity stability

1. Introduction

In the one-step preparation of binder-containing pigment dispersions, relatively low dosages in the range of 1 – 3 % of wetting and dispersing additive(s) can produce impressive results. However, there are limitations. One of the major limitations of the more common carboxyl-functionalised acrylic and styrene-acrylic binders in solution is the limited compatibility of the resulting pigment dispersions with a wide range of letdown binders. To overcome such shortcoming(s), one approach is to first prepare binder-free pigment dispersions followed by a second stage in which the pigment dispersion is converted into a finished ink by incorporation of polymeric binder(s). From the point of view of the

applications of ink, a stable state of dispersion of the pigment is in any case necessary to achieve the desired properties (Atamny et al., 1992, Bourrat, 1993, Ehrburger-Dolle et al., 1994) (Lin, 2003).

In the preparation of binder-free pigment dispersions, the aim is almost always to furnish an adequate amount of stabilising dispersant molecules (Wang et al., 2009) (Yoon et al., 2004). Furthermore, the dispersion formulation is designed to perform suitably on the dispersion preparation machinery with little or no direct consideration for the required properties in the finished ink (Schak, 1997) (Zois et al., 2001). However, the finished ink is formulated to meet a completely different set of requirements such as printability, adhesion to the substrate, resistance to the action of water and/or chemicals and so on (Thompson, 1995) (Frimova et al., 2006) (Merilampi et al., 2009). Consequently, in the two-step ink preparation process, the amount of binder solids that is incorporated in the letdown stage is significantly higher than that in the pigment dispersion. In an extreme case, as in the present study, the binder is not present at all in the pigment dispersion formulation. Thus in such cases, the amount of binder required to achieve the desired properties of the finished ink is added only in the let-down stage. This significant change in composition from the binder-free pigment dispersion to the let-down mixture can result in 'pigment shock'. Pigment shock can occur when a pigment dispersion containing no/low binder solids is formulated into an ink using a let-down mixture which has very high binder solids content. In this situation, a difference in osmotic pressure of the solvent exists in the dispersion and in the let-down mixture. Due to the difference in the binder-solvent ratio, the let-down mixture can draw solvent molecules rapidly from the pigment dispersion, thus forcing the pigment particles to flocculate (Goldschmidt and Streitberger, 2003).

Flocculation, which occurs as a result of pigment shock, results in an increase in the average particle size and accelerated sedimentation (Goodwin and Ottewill, 1991) (Smolarek et al., 2012). Thus, particle size distribution analysis (Frimova et al., 2006) and sedimentation analysis (Tay and Edirisinghe, 2002) can be used to estimate dispersion stability. Techniques based on multiple light scattering (MLS) can also be employed to optically detect any changes in the particle size during aging tests. Since aging is an exothermic process, thermal analysis techniques, such as Differential Scanning Calorimetry (DSC) can be employed to predict the long term stability (aging properties) of an ink. Monitoring of rheological characteristics versus time can also provide insight into the stability of dispersion/ink. A stable viscosity profile over an extended time period can be regarded as an indication of the ink stability (Mewis and Haene, 1993).

In this study, the letdown stability of dispersions of non-printing ink grades of carbon black pigment was analysed. For this, the optimised binder-free pigment dispersions, that were prepared following a 4-step method (Ali and Lin, 2018) (Ali et al., 2019), were formulated into finished inks containing 100%, 150% and 200% 'binder on the weight of pigment' (%BOWP). To assess the let-down stability, the viscosity profiles of the formulated inks were recorded and analysed for any signs of pigment aggregation. The screen printability of the suitably performing inks was also tested by printing the inks on textile substrates using a lab-scale screen printer.

In applications such as printing of functional inks, the integrity of the final ink deposit is as important as the intrinsic functionality of the ink. This is because a properly formulated ink is not likely to yield the desired functionality after printing if the final ink layer structure is disintegrated and is characterized by the presence of several discontinuities. In the case of printing of functional inks on textile substrates, the ink film integrity is of a greater concern compared to when such functional inks are printed on other flexible substrates such as plastics, etc. This is because among the class of flexible substrates, textile fabrics are known to have a greater extent of surface roughness and structural porosity, generally. Coating a textile fabric with a suitable primer coating results in a smoother surface for printing (Park et al., 2007). These effects occur due to the events that occur with such a primer layer. The first of these concerns the blocking and/or covering of the pores and capillaries in the fabric structure. This is particularly important in case of woven fabrics in which pores can run through the thickness of fabric. The second involves the masking of the short fibres and other irregularities on the surface of the substrate, thus reducing the overall roughness of the surface. Owing to the aforementioned concerns in functional printing on textile substrates, it was considered important in the present study to analyse the integrity of films that were deposited on woven fabric substrates using the inks that possessed satisfactory intrinsic characteristics such as stability.

2. Materials and Methods

2.1 Materials

Table 1 contains the details pertaining to the materials that were used to prepare pigment dispersions as reported in another study by the authors (Ali et al., 2019). It is imperative to mention here that both Ensaco 250G (BET surface area 62 m^2/g) and Ensaco 350G (BET surface area 840 m^2/g) are non-printing ink grades of carbon black and are characterized by very low volatile matter content, ca., 0.2 – 0.3%. The binders that were used for formulation of the finished inks are listed in Table 2. For screen printing, a binder coated, 100% polyester plain woven fabric was used as a substrate while for the analysis of ink film integrity, uncoated and binder-coated 100% cotton and 100% polyester plain woven fabrics were used, as elaborated in Section 2.4.

Table 1: Dispersion formulations considered for let-down stability testing

S. No	Dispersion formulation		Dispersion symbol
	Pigment (symbol), wt%	Dispersant (symbol), %DOWP[1]	
1	Ensaco 250G (C2), 23%	Solsperse 44000 (D1), 17.5%	C2D1
2	Ensaco 250G (C2), 23%	BYK-190 (D2), 17.5%	C2D2
3	Ensaco 250G (C2), 23%	Tego Dispers 760W (D3), 17.5%	C2D3
4	Ensaco 350G (C3), 11%	Solsperse 44000 (D1), 155%	C3D1
5	Ensaco 350G (C3), 11%	BYK-190 (D2), 155%	C3D2
6	Ensaco 350G (C3), 11%	Tego Dispers 760W (D3), 155%	C3D3
[1]Dispersant amount on the weight of pigment			

Table 2: Binders used for letdown of pigment dispersions into finished inks

S. No	Binder	Symbol
1	Impranil DLC-F	B1
2	Impranil LP GHG 519	B2
3	Printofix Binder 83	B3

2.2 Let-down procedure to formulate finished inks

To identify any incompatible binder-dispersant combinations, compatibility tests were carried out before the pigment dispersions were let-down with binders. For this purpose, various amounts of binder(s) and dispersant(s) were mixed together and visually assessed for any signs of binder-dispersant incompatibility, such as precipitation of either the dispersant or the binder or both. The mixtures were also deposited onto glass slides and qualitatively analysed for the film opacity, cloudiness, clarity, adhesion and cracking of film after air drying for 48 hours at ambient temperature and also after heating at 70 °C for 1 hour. This approach provided confidence in establishing that the rheological profiles of the finished ink that were recorded after incorporation of a binder in a dispersion were not influenced by any potential incompatibility in any given binder-dispersant combination.

For the aforementioned binder-dispersant compatibility testing, solutions of 10 wt% of various dispersants were mixed with suspensions of 30 wt% and 50 wt% of various binders. On the other hand, solutions of 40 wt% of various dispersants were mixed with suspensions of 10 wt% and 20 wt% of various binders. The selected dispersant loading and binder loading for preparation of mixtures for binder-dispersant compatibility tests represented most of the inks that were formulated to contain 100% 'binder on the weight of pigment' (referred to as %BOWP).

For the formulation of a finished ink, 10 g of a pigment dispersion was weighed in a 25 mL beaker and the required amount of binder was added drop-wise using a pipette while the mixture was continuously stirred using a magnetic stirrer. The beaker containing the ink was sealed using parafilm to avoid evaporation and mixing was continued for 10 minutes after addition of the required amount of the binder. The ink was then collected in a glass vial that was sealed with a PTFE sealed cap to avoid exposure to the air and to prevent evaporation of water. Each of the pigment dispersions, listed in Table 1, was let-down with binders B1, B2 and B3. The inks were formulated to contain 100%, 150% and 200% BOWP), the relevant calculations for this are provided in Table 3 and Table 4 for Ensaco 250G (C2) and Ensaco 350G (C3), respectively.

Table 3: Let-down of pigment dispersions containing 22 wt% Ensaco 250G (C2).

	Formulae	**Binder solids (%BOWP)**		
		100%	**150%**	**200%**
Dispersion amount (g)	A	10	10	10

Pigment content (g)	B = 0.23 x A	2.3	2.3	2.3
Binder, 100% solids (g)	C	2.3	3.45	4.6
Binder, 40% solids (g)	D = (C x 100)/40	5.75	8.62	11.5
Amount of ink (g)	E = A + D	15.75	18.62	21.5
Pigment loading in ink (wt%)	F = (B/E) x 100	14.603	12.35	10.69

Table 4: Let-down of pigment dispersions containing 11 wt% Ensaco 350G (C3).

	Formulae	**Binder solids (%BOWP)**		
		100%	**150%**	**200%**
Dispersion amount (g)	A	10	10	10
Pigment content (g)	B = 0.11 x A	1.1	1.1	1.1
Binder, 100% solids (g)	C	1.1	1.65	2.2
Binder, 40% solids (g)	D = (C x 100)/40	2.75	4.125	5.5
Amount of ink (g)	E = A + D	12.75	14.125	15.5
Pigment loading in ink (wt%)	F = (B/E) x 100	8.627	7.787	7.096

For rheological analyses, a TA Instruments AR-1500 EX rheometer with a cone-and-plate configuration, was employed. The change in viscosity profile after the incorporation of a binder was recorded and compared against the viscosity profile of the corresponding binder-free pigment dispersion. The inks were stored in sealed vials at ambient temperature (22 – 25 ^{o}C) and the viscosity profile of an ink was also recorded after 4 weeks of storage.

2.3 Screen printing of formulated inks

A Rokuprint SD05 screen printer, with a flat screen attachment was used in the experiments to test the printability of the inks that were selected after letdown stability testing. During printing, the samples were held down using the vacuum plate attachment provided with the screen printing machine. The printing parameters were set on the basis of the results of a separate study by the authors (Muhammad Ali et al., 2019).

A square-edged polyurethane squeegee of 80 shore hardness was used to draw the inks across the screen. A stainless steel plain woven mesh screen having 32 microns thread diameter and 56 microns aperture size was used and the snap-off height was set at approximately 1 mm. The stencil emulsion thickness was approximately 20 microns. The design that was printed was in the form of a 1 mm wide, 50 mm long line. This was followed by evaluation using a Keyence VHX-2000E digital optical microscope in order to establish the quality of prints in terms of print registration quality and the integrity of ink film deposits.

2.4 Analysis of Ink film integrity

Testing of the finished inks on different commonly used textile substrates, i.e., 100% cotton and 100% polyester plain woven fabrics, with and without a binder primer layer, was

considered to be important to establish the general applicability of the conclusions drawn from this study. In a separate set of screening experiments, B3 was identified as the most suitable binder for primer coating in terms of the structural features of the primer layer and its durability to withstand washing. The formulated inks were deposited on uncoated and binder-coated, cotton and polyester woven fabric substrates, followed by analysis of the ink film integrity using a Keyence VHX-2000E digital optical microscope.

3. Results and Discussion

3.1 Let-down stability of the C2 pigment dispersions

In this section, the results of viscosity stability analyses of the inks prepared from the dispersions of C2 pigment are discussed with respect to the three binders that were used to formulate finished inks. As shown in Figure 2(c,d), a considerable change in the viscosity occurred in the inks prepared by adding B2 in the C2D1 dispersion. Furthermore, deviation from shear-thinning rheology was also recorded for these inks. The above mentioned changes in viscosity were more pronounced in the ink containing 200% BOWP of B2. Repeat viscosity measurements of these inks did not show a consistent trend. However, large variations in viscosity, which were not in-line with the average increase of approximately 11.11% (Figure 1) in the viscosity of binder-free C2D1 dispersion, were recorded in all the repeat measurements. The viscosity of inks prepared by adding B2 in the C2D2 and C2D3 dispersions decreased considerably after four weeks of storage, as shown in Figure 3(c,d) and Figure 4(c,d), respectively. However, in both these cases, the shear thinning rheology was maintained.

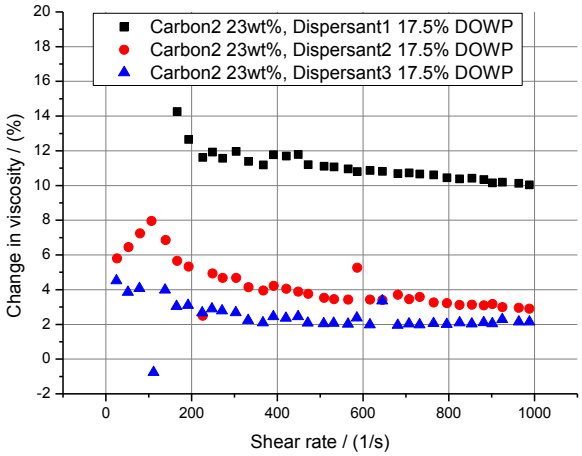

Figure 1: Change in viscosity during four weeks storage of C2 pigment dispersions.

In contrast to the inks of C2 pigment that were prepared using B2, the inks prepared using B1 and B3 were generally very stable as indicated by a small average change in the viscosity after four weeks of storage. The relevant results are provided in Figure 2 – 4 (a,b) and Figure 2 – 4 (e,f). The change in viscosity of most of these inks can be correlated to a

considerable extent with the change in viscosity, as shown in Figure 1, of the binder-free dispersions of C2 pigment.

The viscosity of C2D1 dispersion increased by approximately 11% (SD=0.88) while the increase in viscosity of C2D1B1 inks was between 10-12% (approx.) as shown in Figure 2(a,b). The data shows a relatively weaker correlation between the increase in viscosity of C2D1B3 and C2D1 dispersion. However, this difference is not significant enough to conclude that addition of B3 altered the state of dispersion of C2 pigment. Similarly, as shown in Figure 3, small changes in the viscosity of C2D2B1 and C2D2B3 inks were recorded after four weeks of storage. This is in-line with the increase of 4.2% (SD=1.35) in the viscosity of binder-free C2D2 dispersion. The average increase of approximately 18% (SD=1.22) in the viscosity of ink prepared from C2D2 dispersion and 150% BOWP of B1 was not regarded as a clear indication of B1-induced changes in the state of dispersion of C2 pigment. This is because the viscosity of the other two inks of this series virtually remained unchanged.

The results of viscosity stability analysis, presented in Figure 4, clearly indicated that in terms of viscosity, the C2D3B1 and C2D3B3 inks were fairly stable, as was the binder-free C2D3 dispersion for which the average increase in viscosity was 2.4% (SD=0.88). In summary, the results of viscosity stability analysis of the binder-free dispersions of C2 pigment and the finished inks prepared thereof from these dispersions were generally in good agreement and indicated that these formulations were very stable. The inks prepared by adding B2 are an exception.

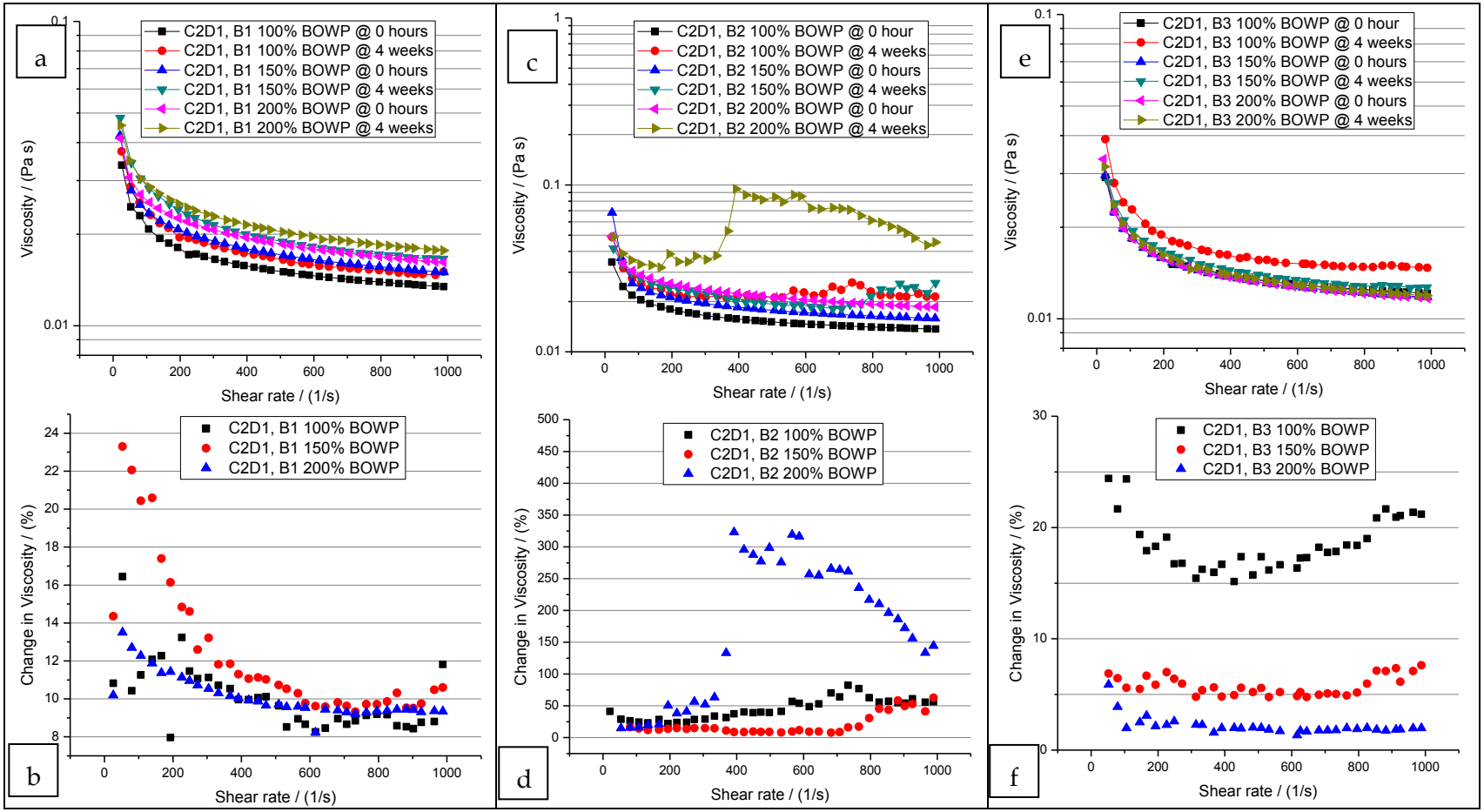

Figure 2: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C2D1 dispersion.

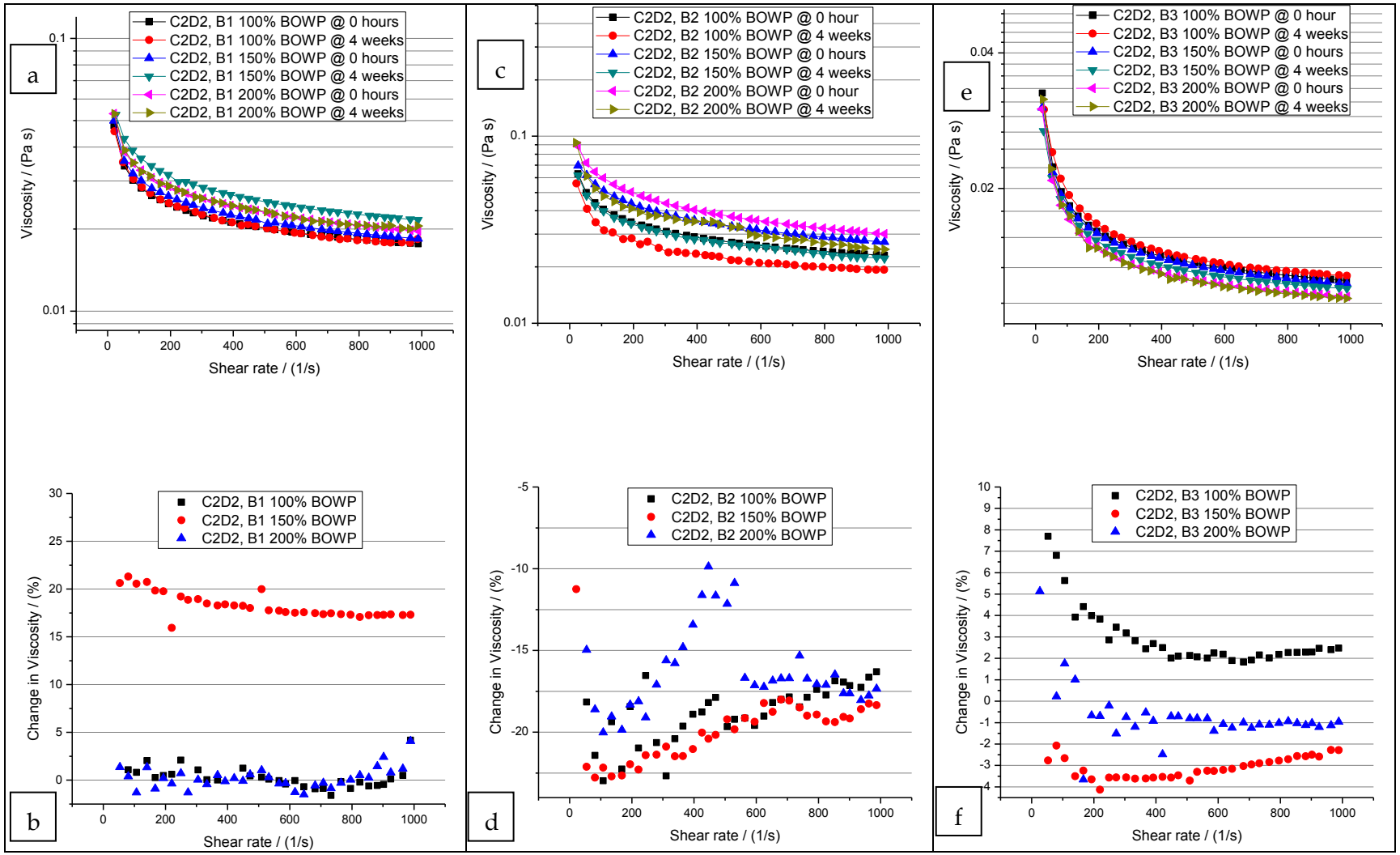

Figure 3: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C2D2 dispersion.

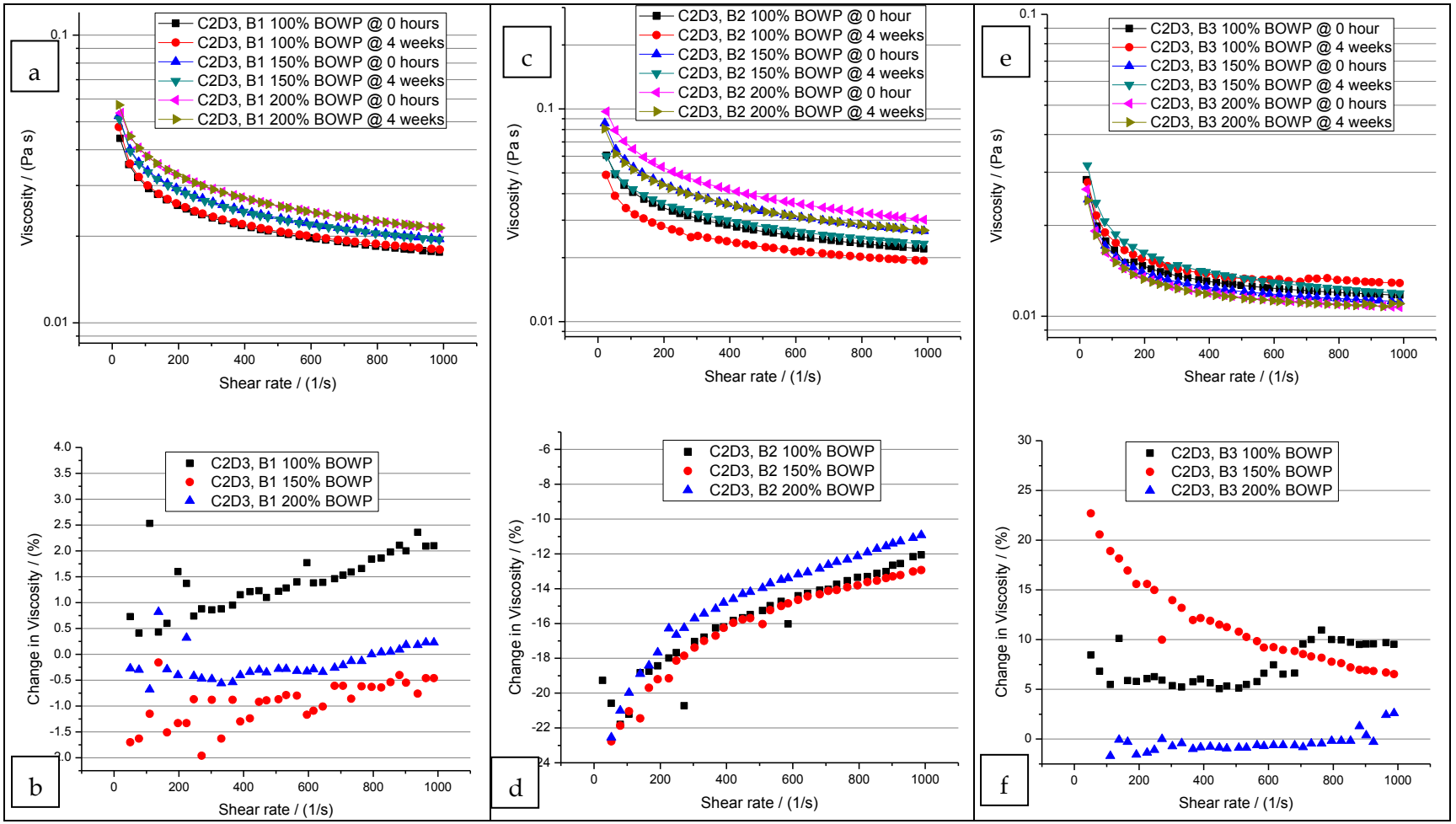

Figure 4: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C2D3 dispersion.

3.2 Let-down stability of the C3 pigment dispersions

The viscosity of the inks prepared from C3D1 dispersion increased considerably after four weeks of storage. The relevant data is provided in Figure 6 which also depicts that the increase in viscosity was smaller at higher shear rates, generally. As depicted in Figure 5, a very similar trend of increase in the viscosity of binder-free C3D1 dispersion was recorded with the average increase being 42% (SD=8.89). This is in good agreement with the increase in the viscosity of the inks that were prepared from C3D1 dispersion (the exception is the ink prepared by adding 100% BOWP of B3). However, the data did not provide a clear indication of a correlation between the amount of binder in the ink and the change in viscosity after storage.

In contrast to the inks prepared from C3D1 dispersions, the inks prepared from the C3D2 and C3D3 dispersions possessed considerably stable viscosity during storage, as shown in Figure 7 and Figure 8, respectively. This is in-line with the results of viscosity stability analysis of the C3D2 and C3D3 dispersions, which showed that virtually no change occurred in the viscosity of these dispersions after four weeks of storage. The average increase in the viscosity of the two dispersions is tabulated in Table 5. Little or no change in the viscosity or no deviation from shear thinning rheology was recorded, particularly for the inks prepared by adding B1/B3. The only exception was the ink containing C3D3 dispersion and 100% BOWP of B3, for which the average increase in the viscosity after storage was 46% (SD=1.17). This was not regarded as an indication of instability of the C3D3 dispersion after let-down with B3. This is because the viscosity of the other two inks of this pigment-dispersant-binder series virtually remained unchanged.

Referring to Figure 7(c,d) and Figure 8(c,d), the increase in viscosity of inks that were prepared by adding B2 in the C3D2 and C3D3, was relatively greater compared to the increase in the viscosity of the relevant binder-free dispersions (data provided in Table 5). However, the difference is not large enough to establish that B2 significantly altered the state of pigment dispersion in these formulations.

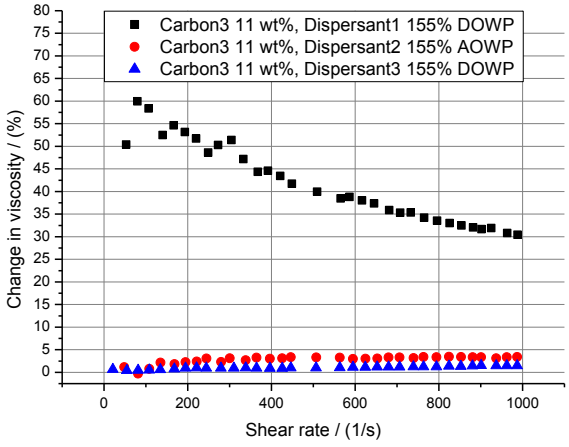

Figure 5: Change in viscosity during four weeks storage of the C3 pigment dispersions.

Table 5: Change in viscosity of C3D2 and C3D3 dispersions.

Dispersion composition	Increase in viscosity (%)	
	Average	SD
Carbon3 11 wt%, Dispersant2 155% DOWP	2.79	0.87
Carbon3 11 wt%, Dispersant3 155% DOWP	1.05	0.29

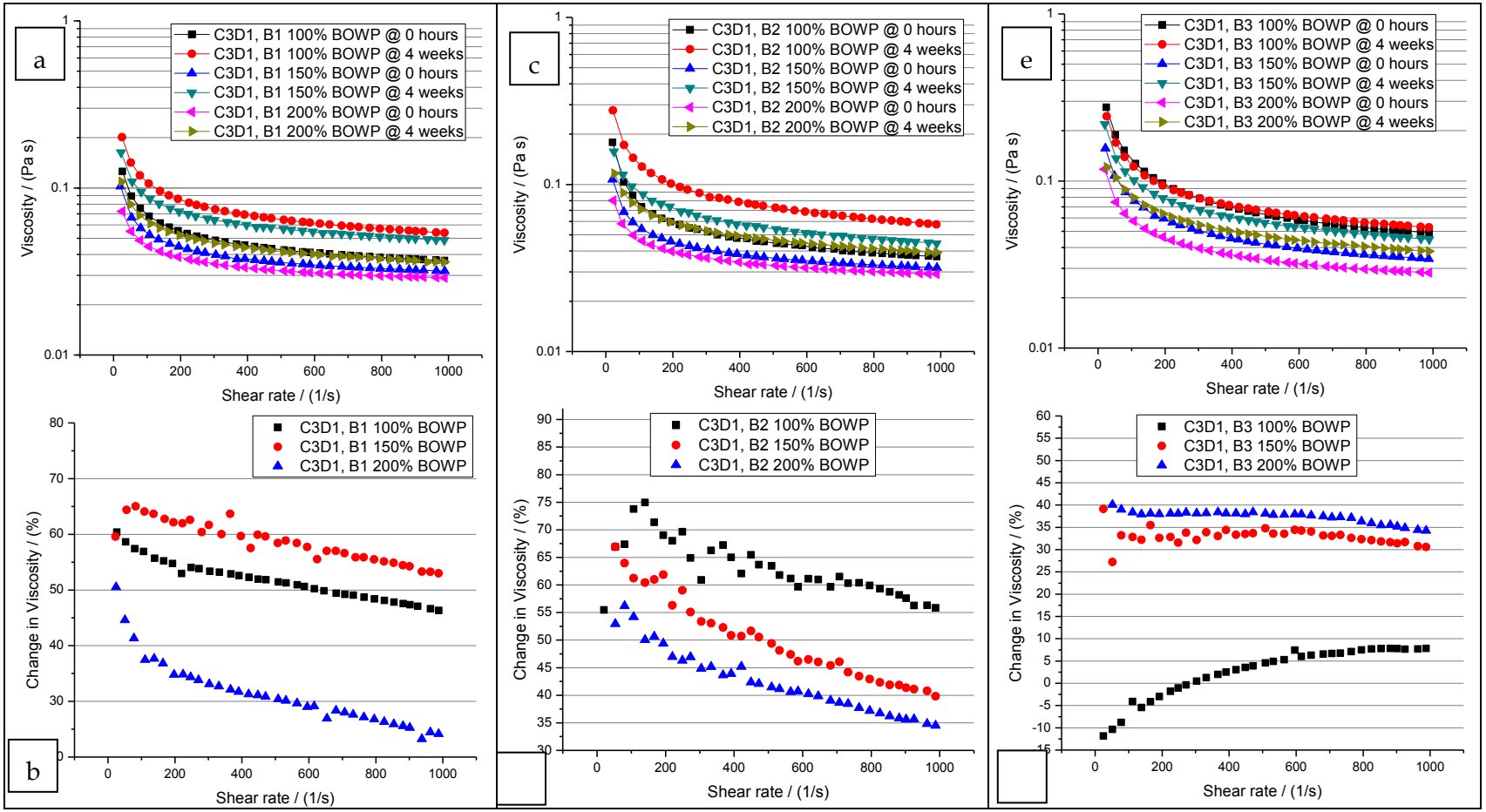

Figure 6: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C3D1 dispersion.

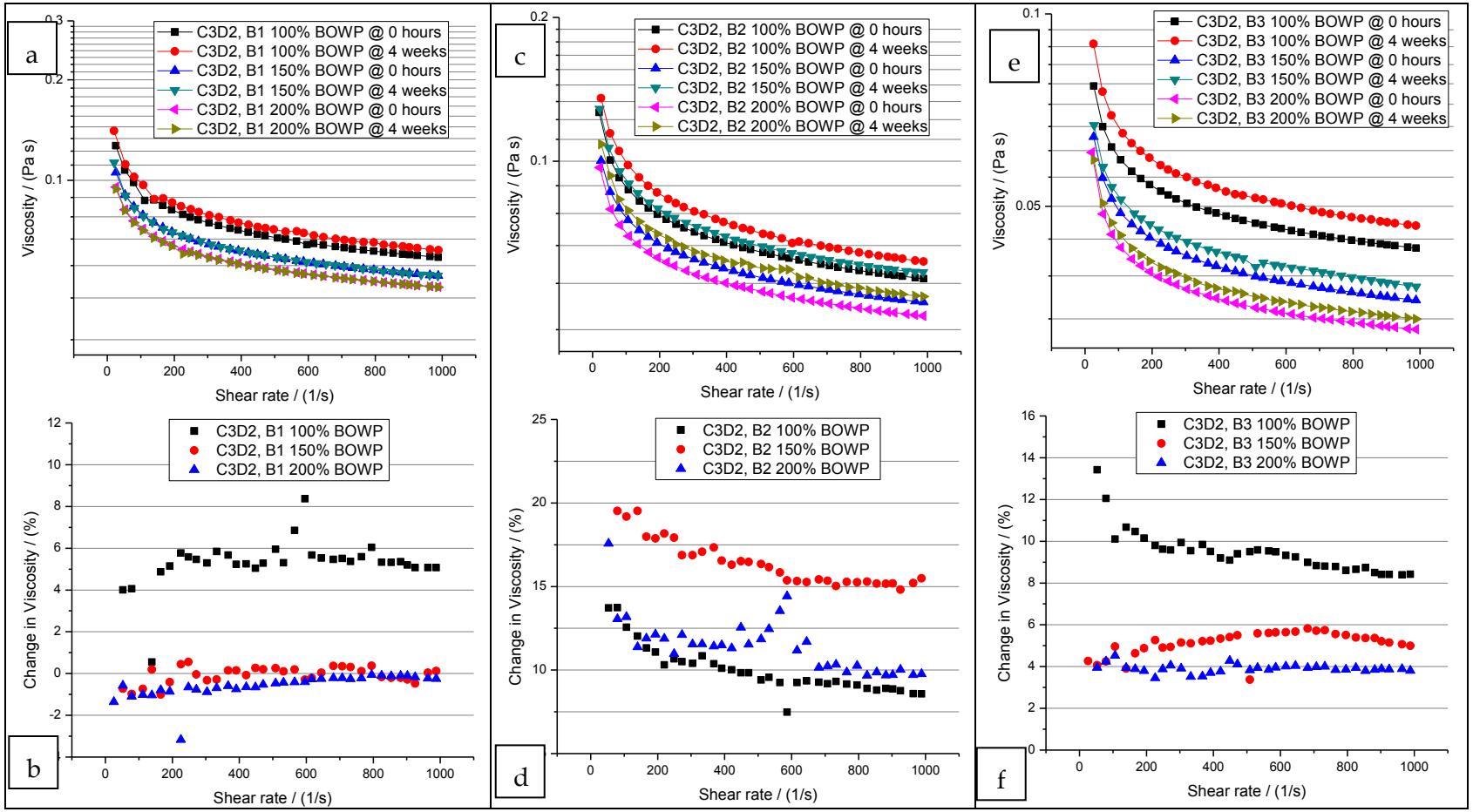

Figure 7: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C3D2 dispersion.

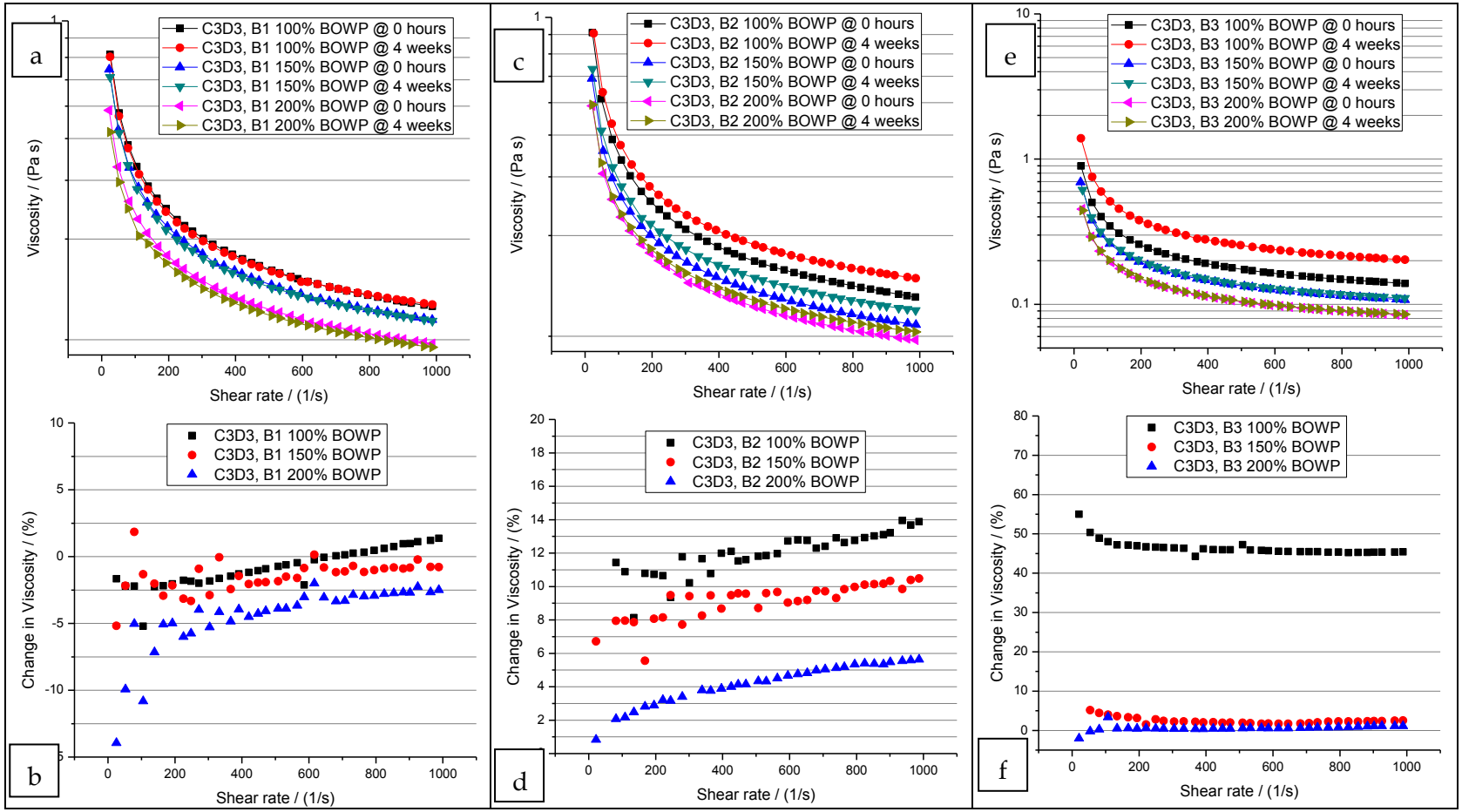

Figure 8: Viscosity stability of inks prepared by adding various amounts of B1/B2/B3 in C3D3 dispersion.

3.3 Screen printability and film integrity of the formulated inks

As evident from the data presented in Figure 2 - Figure 4 and Figure 6 - Figure 8, the inks that were prepared using B3 were relatively more stable compared to the inks prepared using B1 or B2. Furthermore, B3 was found to be better as a primer compared to B1 and B2 and deposition of B1- and B2-containing inks on B3 coated fabrics resulted in cracking of the ink deposit due to difference in the surface energies of these inks and the B3 primer layer. Thus, the inks which were produced using 100 %BOWP of B3 were tested for "screen printability" onto textile substrates. Figure 9 shows the actual dimensions of the 1 mm wide line that was printed using a metal mesh flat screen, as described in Section 2.3. The width of this 1 mm wide line, as obtained by screen printing the inks on coated polyester fabric, was measured while analysing the ink film integrity as discussed in the following section. The images of the prints are provided in Figure 10. Variation in the width of printed lines was considered to be within the acceptable range for such fine lines. The print resolution can be fine-tuned by adjusting the viscosity and/or the printing process parameters. Thus, it was concluded that the screen printability of the inks, formulated from the binder-free dispersions of non-printing ink grades of carbon black, was generally very good. The inks prepared by incorporating 100 %BOWP of B3 were deposited onto uncoated and B3-coated cotton and polyester fabric substrates. As evident from the optical micrographs presented in Figure 11 and Figure 12, all of these inks were found to produce fairly integrated deposits on the fabric substrates that were considered.

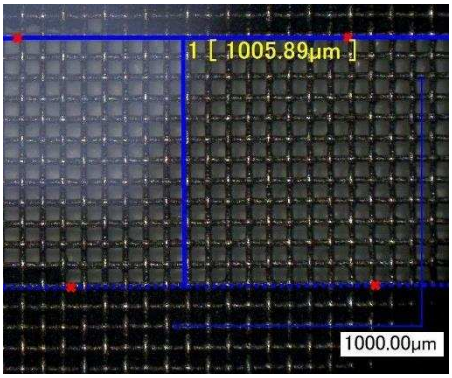

Figure 9: Optical micrograph (Magnification x100) of the stencil used to print 1 mm wide line on fabric substrates.

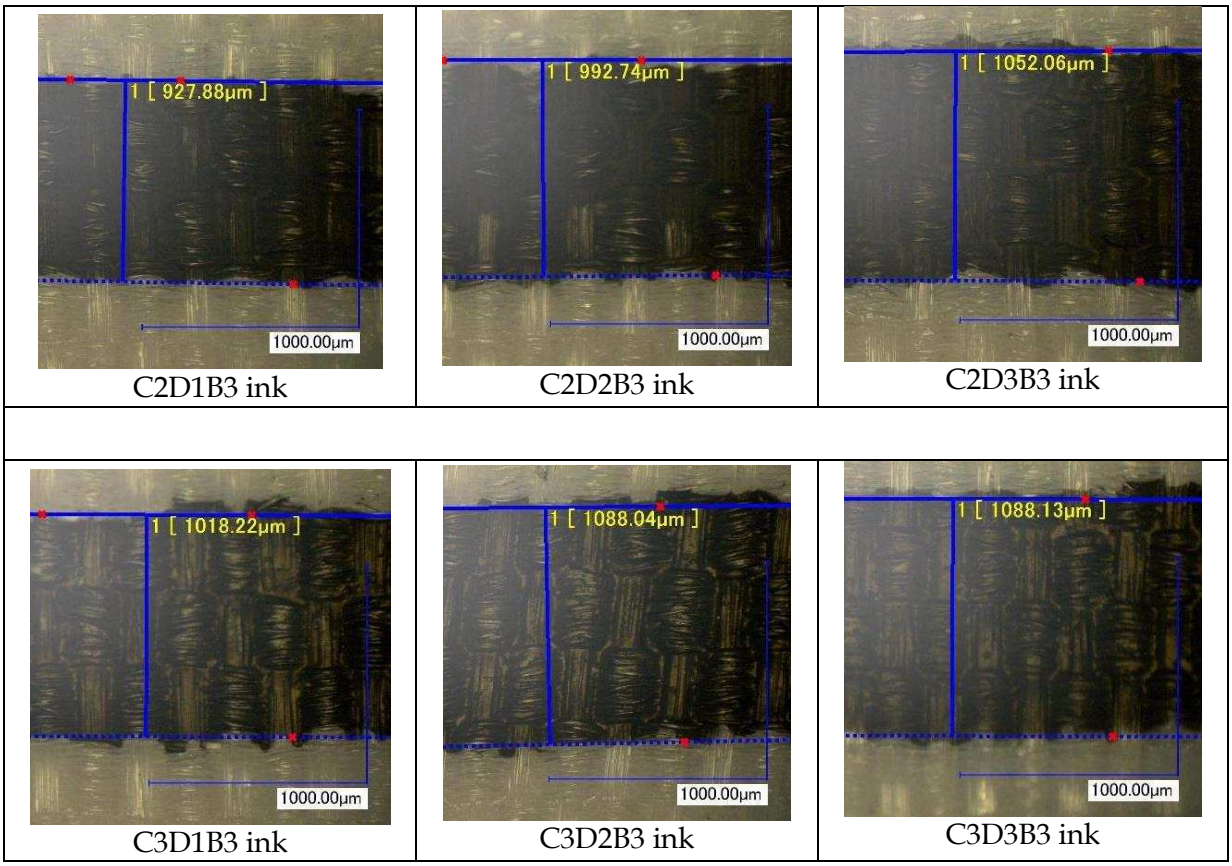

Figure 10: Images of B3-containing inks screen printed onto B3-coated polyester fabric.

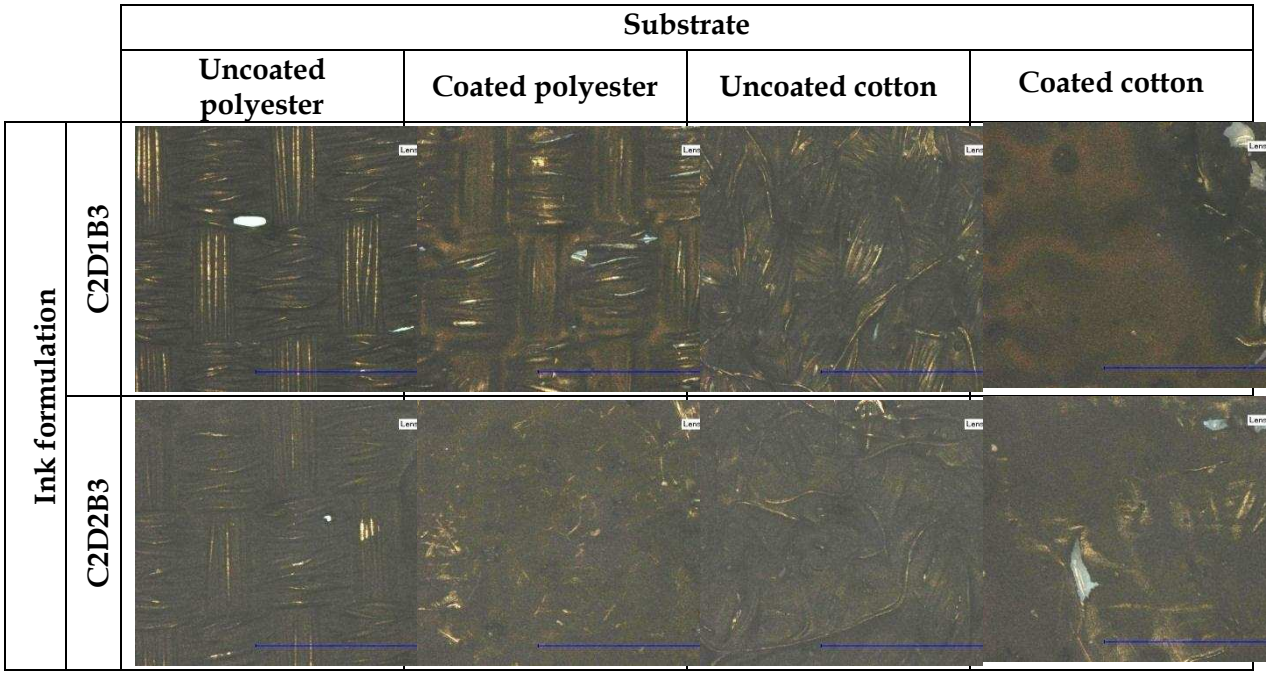

Figure 11: C2 pigment inks deposited onto uncoated and binder-coated cotton and polyester fabric substrates

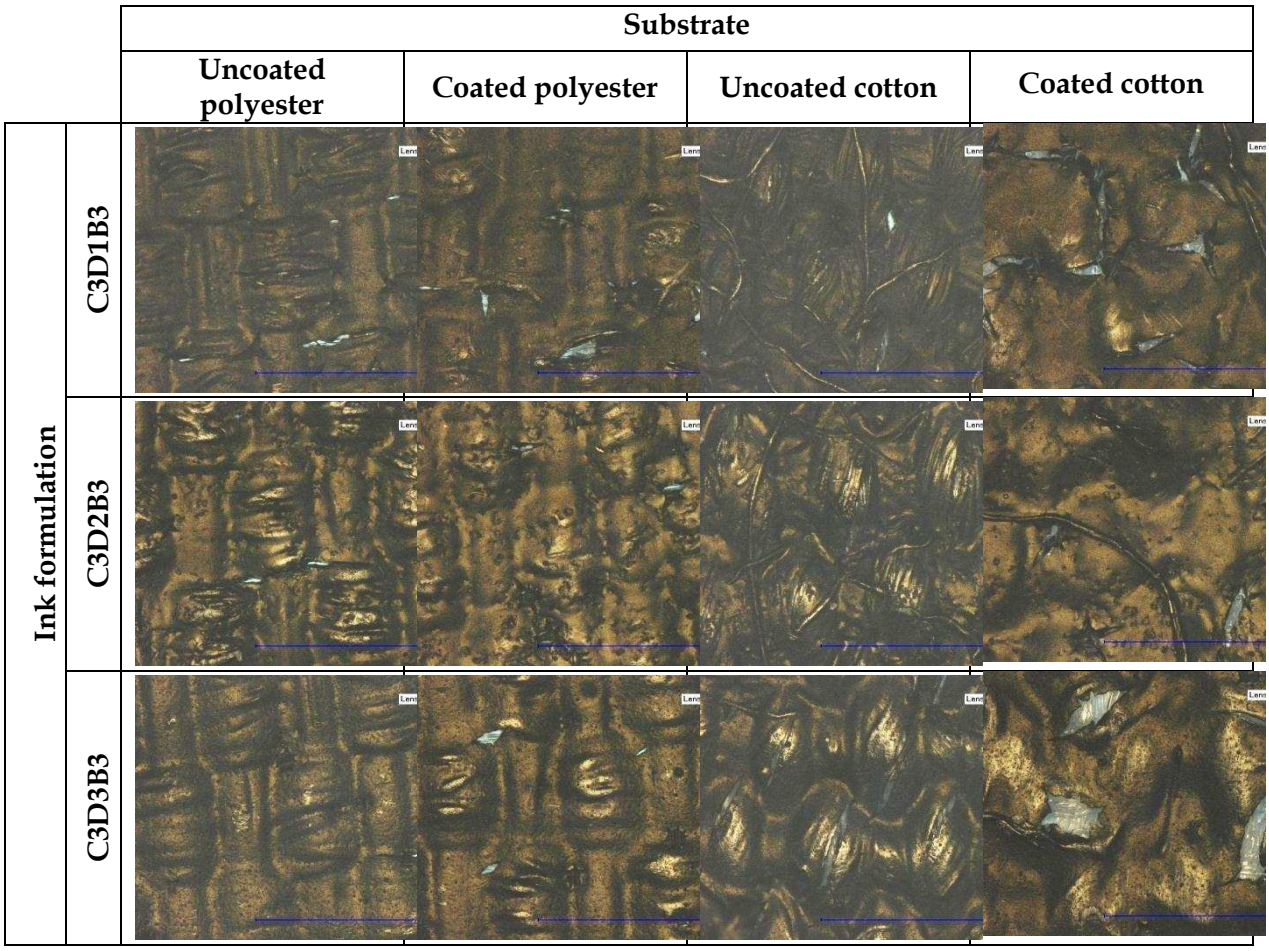

Figure 12: C3 pigment inks deposited onto uncoated and binder-coated cotton and polyester fabric substrates

4. Conclusions

In the present study, we have demonstrated that waterborne, binder-free dispersions of non-printing ink grades of carbon black pigment can be converted into stable finished inks using a various amounts of different binders. The results validate that fact the binder-free dispersions of low surface area carbon black pigment as well as high surface area carbon black pigment that are produced by following an optimisation procedure, as outlined by the authors in a previous study, can be converted into stable finished inks that are suitable for application on textiles by methods such as screen printing. The rheological profiles recorded after incorporation of binders and after four weeks of storage clearly indicate a shear thinning behaviour of the inks with virtually no signs of pigment aggregation. Thus, it can be said that the inks possessed a considerable shelf-life stability as well. Furthermore, the screen printability of inks onto textile substrates was also found to be satisfactory as indicated by the quality of pattern registration. The inks that were found to be stable upon storage were also able to produce ink layer deposits with an integrated structure on uncoated and binder-coated woven textile substrates. These findings are envisaged to be useful in providing an insight into the printing of functional inks onto textile substrates – a demanding aspect in the field of e-textiles.

5. Acknowledgements

The authors would like to acknowledge NED University of Engineering & Technology, Karachi, for providing the financial support to conduct this research. The authors would also like to acknowledge TIMCAL (now Imerys Graphite & Carbon), Evonik Industries, BYK Chemie, Clariant (now Archroma) and BASF (UK) for kindly providing the materials that were used in this study.

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