Digital holographic interferometry and dynamic speckle as methods to determine the drying time of paints

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Abstract Drying time of different types of paints is an important parameter to be determined since it serves to improve production and manufacturing processes. The development of a good quality control, allowing evaluating if the paint has achieved optimum characteristics after being applied, would aid to make corrections before final application in order to obtain the desired results. Both digital holographic interferometry (DHI) and dynamic speckle are suitable techniques to accurately assess drying time of paints. In DHI a sequence of interferograms, taken at regular time intervals, allows observing the evolution of the drying process by detecting shape variations at interferometric scales (i.e. at scales in the order of the wavelength used). A relatively simple off-axis Fourier lensless holographic arrangement and suitable numerical reconstruction and analysis algorithms can be used to determine the drying time on small samples. In turn, the dynamic speckle method allows measuring the drying time by evaluating the speckle mobility of the fluid as it dries. This technique requires an even simpler experimental arrangement, with a low-resolution camera (1 Mpix) and a semiconductor laser whose wavelength must be chosen to be compatible with the color of the paint. In this work we present a comparative analysis, between DHI measurements and dynamic speckle methods, to determine the drying time of different types of paints. These results were also compared to the method that is currently used in paint manufacturing process. The results obtained show that both techniques are reliable tools to evaluate the drying time of paints, which is an interesting parameter for improving the paint manufacturing process.

Keywords: Holographic Interferometry, Dynamic Speckle, Paint, Drying Time

1 Introduction

The paintings are materials manufactured as fluid but that during use must be transformed into solids. Initially, during the production process the paint must behave like a fluid, with all the desirable characteristics of this matter state. However, the paints are defined as not Newtonians fluids being its viscosity variable with temperature in a non-linear way. This fact adds complexity to their manufacture.
Paints manufacture process, involves a series of steps such as mixing and pigmentation or packaging and conservation in controlled ambient condition. The basic pint components can be listed as:

Base
Pigments
Binders
Solvents
Plasticizers
Loads

Each formula includes a series of steps in which these components are added in liquid or solid state.
Test of quality control concerning viscosity with rotational viscometers and drying time are usually performed during the manufacturing process. The first one during different stages of formula, the latter once the production process ends.
When the paint is applied over de surface solidifies trough the so–called curing process which comprise two stages drying and hardening.
The drying stage corresponds to the evaporation of solvents and diluents, added reduce its viscosity.
Hardening stages corresponds to process of entanglement of the polymeric chains which form the paint base.
The measurements of the drying time used to be fairly elementary and shaped subjective depending on the ability of the lab technician. Generally it is used a normalized surface in which the paint has been applied

with the help of a spreader to generate paint layers of known thickness. The lab technician defines the drying time according to the stickiness of the painted surface. Usually ranging between 2 or 4 hours, then the formula is validated and the packaging process is authorized.
In high production cycles, this represents an important dead time, in which the painting should be maintained on the production batch. In addition, solvents must be added to avoid the drying of the outer layer to the paint (formation of “milk cream”).
Several works have been published regarding the monitoring and control of the paints drying process. Most of them concerning dynamic speckle techniques, like speckle interferometry [2][3] or dynamic speckle correlation [4][7]. In turn, in a small proportion, other authors have implemented methods based on digital holographic interferometry (DHI) [1][8]. Dynamic speckle methods represent a very straightforward alternative to assess drying time of paints, since the experimental setup is quite simple and speckle activity is a direct qualitative method to inspect the evolution of the drying process. As a counterpart, the high sensitivity of DHI techniques makes the observation and analysis of paint drying somewhat more difficult. However, DHI allows inspecting in a very precise manner the last stages of drying and also provides quantitative values to determine variations in the thickness of the coatings.
In this work we have analyzed the drying of a commercial paint, from Sorbalok S.A®, by means of dynamic speckle and DHI simultaneously. The possibility of using both techniques to monitor the drying process time is discussed. Particularly dynamic speckle offer a simple way to estimate the time of drying while DHI allows us to follows the evolution of the dried.
This method may diminish significantly the time consumed in production plants and improve the quality control in an objective way.

2 **Theoretical background**

2.1 Dynamic speckle

Speckle patterns are formed when objects are illuminated with a coherent light source and possess information about the object's surface at a scale above of the wavelength used [9]. The variation, or evolution, of a pattern with time, i.e. a dynamic speckle pattern, allows further analysis of the object in the temporal domain. The degree of activity is what we call the speckle activity (SA).
SA was analyzed through a method that measures the average fraction of pixels whose intensity changes as a function of time (from image to image) in more than a certain amount, called speckle noise. This method is described in detail in Ref. [10] and here we limit to show the basic equation to determine the SA of a sequence of images, which reads

$$SA = \frac{1}{n\times m}\sum_{k=1}^{N-1}\frac{1}{N-1}\sum_{i=1}^{m}\sum_{j=1}^{n}\varphi[|I_{k+1}(i,j) - I_k(i,j)| - r] \quad (1)$$

where N is the number of images from which speckle activity should be determined, m and n are the image sizes, the scaling factor (m,n) transforms the obtained quantity to the desired area fraction of varying pixels and φ(x) is the well-known Heaviside function. Equation (1) allows calculating the SA from a sequence of images, extending through a certain elapsed time. Therefore, there are basically three important parameters that must be taken into account for the analysis, namely: (i) the number of frames or images, $N\geq 2$ usually $N=10$, used to calculate a single SA value, (ii) the period of time between those N frames, τ, whose lower limit is given by the highest attainable acquisition rate of the camera and (iii) the period of time, t, between subsequent calculations of SA. Hence, each calculated value is performed over a finite temporal extension, between the firsts and the last frame, given by$\Delta\tau = [N-1]\tau$. This is schematized in Fig. 1.

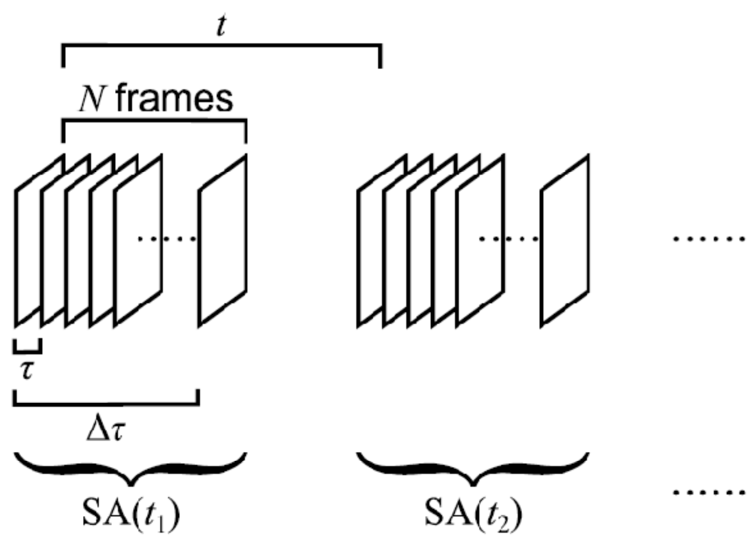

Fig.1.Schematic representation of different parameters involved in the acquisition and calculation of speckle activity evolution.

2.2 Digital holographic interferometry

A digital hologram is a digitized image (i.e. acquired by digital CCD or CMOS cameras) of the unfocused interference map between two coherent wavefronts, namely: (i) a reference wave impinging directly onto the digital sensor and (ii) a wavefront that is scatter from the surface of the object under analysis, called the object wave. The hologram contains information about the object physical three-dimensional shape, which can be reconstructed by numerical methods to get the intensity and relative phase of waves emerging from each point of the object [11]. DHI consists in the interferometric comparison of numerically reconstructed phase information from (at least) two digital holograms of an object in an (1) undeformed (reference) state and (2) a deformed state [12], whenever the deformation is greater few wavelengths.
Considering the Fresnel approach of light propagation and a typical off-axis lensless digital Fourier holographic arrangement, as shown in Ref. [13], each hologram is reconstructed by means of a single fast Fourier transform (FFT) and complex-matrix multiplication, as

$$H_{1,2}(n,m) = C_{1,2} e^{\frac{i\pi}{\lambda d}(\xi^2+\eta^2)} F^{-1}\left[h_{1,2}(x,y)\right] \quad (2)$$

Here, subscripts 1 and 2 correspond to each state, (ξ,η) are the coordinates on the object plane, (x,y) are the coordinates on the hologram plane, $h_1(x;\ y)$ and $h_2(x;\ y)$ are the holograms at each state, C_1 and C_2 are complex constants, λ is the wavelength used for hologram recording, d is the distance between the object and the hologram planes and F^{-1} stands for a two-dimensional inverse FFT. The constant and exponential factors in front of the FFT can be disregarded for a DHI analysis [14]. Thus, $H_1\ (\xi,\eta)$ and $H_2\ (\xi,\eta)$ describe the complex wavefronts at each state of the object, propagated back to the object plane.
The relevant parameter in DHI measurements is the phase difference $\Delta\varphi(\xi,\eta)$ between both states of theobject, which can be recovered from (2) by means of

$$\Delta\varphi(\xi,\eta) = tan^{-1}\left[\frac{Im\left(H_2(\xi,\eta)H_1^*(\xi,\eta)\right)}{Re\left(H_2(\xi,\eta)H_1^*(\xi,\eta)\right)}\right] \quad (3)$$

where the asterisk (*) denotes complex conjugation. This operation yields a map of phase difference values in the range [-π, π], which is called the wrapped interference phase map (or interferogram)[14].
Usually, subsequent filtering and unwrapping processes must be performed to describe the actual object deformation with respect toits original state. Filtering operation is effectively achieved through a sine/cosine average filter [14] while there areseveral methods to perform one- or two-dimensional phase unwrapping.

The phase difference matrix $\Delta\varphi(\xi,\eta)$ obtained numerically from Equation (3) is proportional to the displacement or deformation of each point of the object between states, which is expressed mathematically as

$$\Delta\varphi(\xi,\eta) = \vec{S}(\xi,\eta) \cdot \vec{d}(\xi,\eta) \tag{4}$$

The vector $\vec{S}(\xi,\eta)$ (called sensitivity vector) is determined by the experimental setup and can be adjusted to get maximum sensitivity to displacements or deformations in the desired direction. In turn, the displacement vector $\vec{d}(\xi,\eta)$ is given by the real point-wise deformation of the object and can be calculated from Equation (4) once $\Delta\varphi(\xi,\eta)$ is known.

3 Experimental details

We have analyzed the drying evolution of a red colored synthetic enamel (brand: Sorbalok®, color: orange) bymeans of simultaneous SA and DHI measurements. For this purpose we have implemented a typical Off-axis lensless Fourier digital holographic arrangement with a He-Ne laser light source and a 5 Mpixel CCD camera (Motic 5®). We have added a second 1Mpixel CCD camera (Motic 1000®) with a focusing lens to register speckle images formed directly onto the drying enamel. A simplified scheme of the experimental setup is shown in Fig. 2.
Preliminary experiment of SA performed using a semiconductor laser of 480 nm and paint color "traful" and Motic 1000® camera. In all case more than 2000 pictures was captured for each analysis.
Holograms and SA combined experiences were acquired at intervals of 15 or 60s and speckle images were recorded continuously at intervals (τ) of 1s or 5s. Total observation times elapsed for 2 or 2.5 hours (depending also on each experience), after which we acquire between 480 and 600 holograms and between 7200 and 9000 speckle images. All digitized images were converted to 8 bit gray-scale and processed with algorithms developed under the MATLAB® interface.
For the first series of measurements we have applied the enamel with a nearly rectangular plastic block used as a stamp onto a vertical plain surface. In this way, the enamel owed down the surface due to gravity, providing a painted region with different thicknesses of enamel to observe. For a second experience, we have put a small droplet of enamel on a horizontal surface to avoid owing. In this latter case, we have added an extra mirror to the experimental setup in order to illuminate the object and direct the rejected (object) wavefront towards the camera.

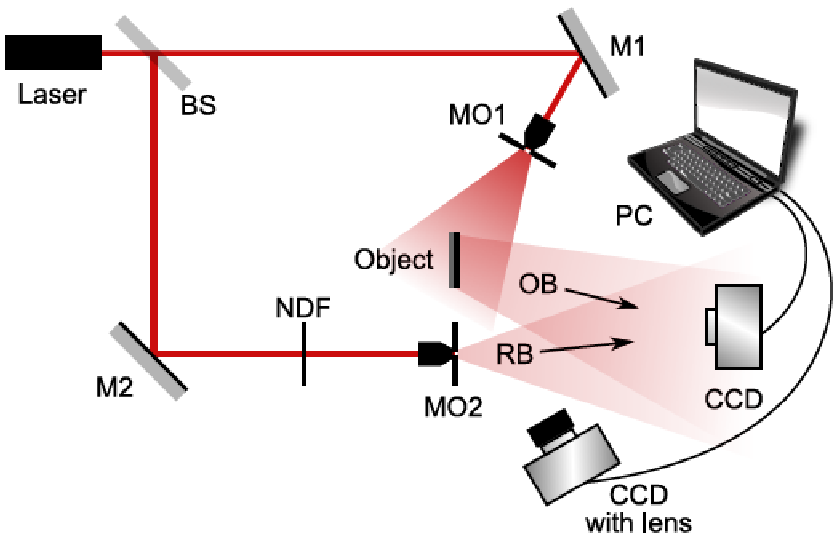

Fig. 2. Simplified diagram of the experimental digital off -axis lensless Fourier holographic arrangement with a second CCD camera with lens to acquire the sequence of speckle images. Laser: He-Ne; BS: beam splitter; Mi: mirrors; NDF: neutral density filter; MO1,2: spatial filters; OB: object beam; RB: reference beam; CCD: digitalcameras.

4 Results and discussion

We present the results obtained from analysis of the data acquired from DHI and SA measurements. It is important to make clear that synthetic enamels have cure a cureing time longer than 8 hours, depending on ambient conditions. However, in what follows we call drying time to the elapsed time the enamel requires to reach a sufficiently steady state after which the paint will not suffer further deformations, as far as the optical techniques like those applied in this work can determine.

4.1 Drying time estimation

A preliminary test was performed to evaluate the relationship between paint quantity and drying time. Two samples with different thickness were measured by SA. A capture period of 30 minutes with 1 second of was defined and the results can be seen in Fig 3.

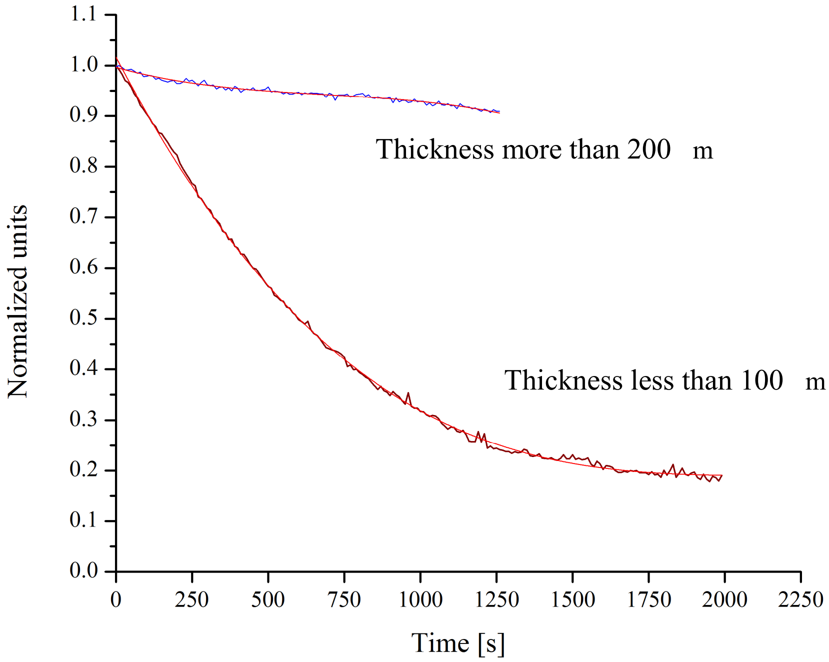

Fig. 3. Evolution of speckle activity obtained by SA on two samples of different thicknesses of the same paint.

As a result of the measurement, it is clear that increasing the thickness of paint increases significantly the drying time. This result indicates that the test sample and the environmental conditions must be constants.
As mentioned before, the firsts experience consisted in observing a region of a vertical surface onto which the synthetic enamel was stamped. Holograms were acquired at 60 s intervals. In Fig 4 we show a reconstructed image of the painted region (left side) and a sequence of selected interferograms showing the evolution of the drying (right side). The enamel was applied on the nearly rectangular region of the vertical surface marked with a solid line and, immediately, it drips in the region marked with a dashed line. As can be observed, the interferograms at the initial stages of drying show a granular speckle pattern in the painted region, without any distinguishable interference fringes. This can be due to the fact that the degree paint deformation between subsequent holograms is much greater than the scale of the wavelength used or because reconstructed wavefronts from subsquent holograms are decorrelated due to a high degree of deformation of the fresh enamel surface.
As the time passes, although no fringes are visible, the interferograms reveal the dried zones (homogeneous gray regions) and other which remains wet (zones with speckle). The last interferogram was the only one of the whole sequence that revealed circular interference fringes, evidencing that the enamel was quite static and drying while reducing its thickness right in the direction pointing towards the CCD camera. At first sight, from this sequence of interferograms, it is neither possible to estimate thickness variations of the

painted region nor a drying time. However, if one calculates the active fraction of the painted area from one interferogram to the other by correctly thresholding the gray values it is possible to obtain depicted in Fig. 5. This curve presents a characteristics sigmoidal decaying shape, which is characteristic of the drying of coatings [2].

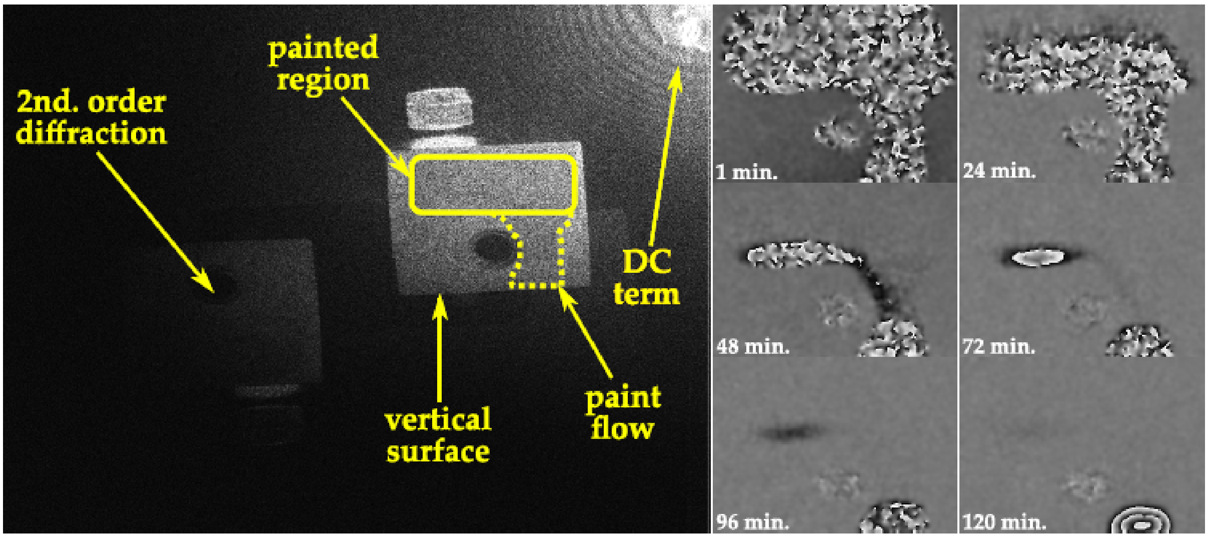

Fig. 4. Reconstructed image of the region under study from a hologram (left side) and sequence of interferogramsobtained during the enamel drying process (right side). The vertical flowing of the enamel can be clearly seen.

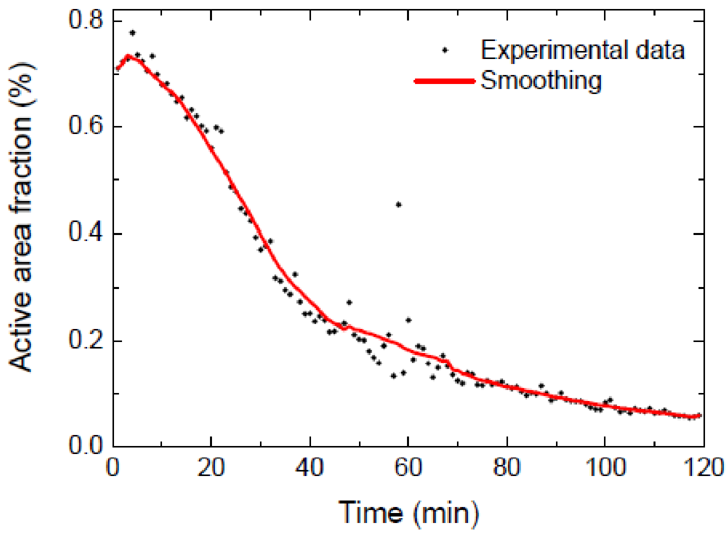

Figure 5. Evolution of active are of dried enamel computed by thresholding adequately the gray values of the interferograms.

In turn, for dynamic speckle measurements of this process, we acquired images at 5 s intervals and the SAcalculations (taking N = 5 frames, which gives $\Delta\tau = 50$ s) showed again a typical decaying sigmoidal curve, as seen in Fig. 6. This curve is smoother than that obtained by DHI and furthermore no smoothing procedure was required. This could be due to the relatively large sampling interval with respect to those generally used for SA measurements and also to diminishing of the noise in SA measurements as compared to DHI ones. The noise level extracted from the processed images is shown as a horizontal dashed line. The inset of Fig. 6 compares the curves obtained with both techniques, SA and DHI, which were normalized for this purpose. From these curvesone can easily estimate a drying time by a non-linear fitting of the data points as they are behind obtained, rejecting the first four or five values of the curves. Therefore, by implementing a suitable fitting procedure there exist the possibility of estimating the drying time of paints during the first 20 minutes of drying.

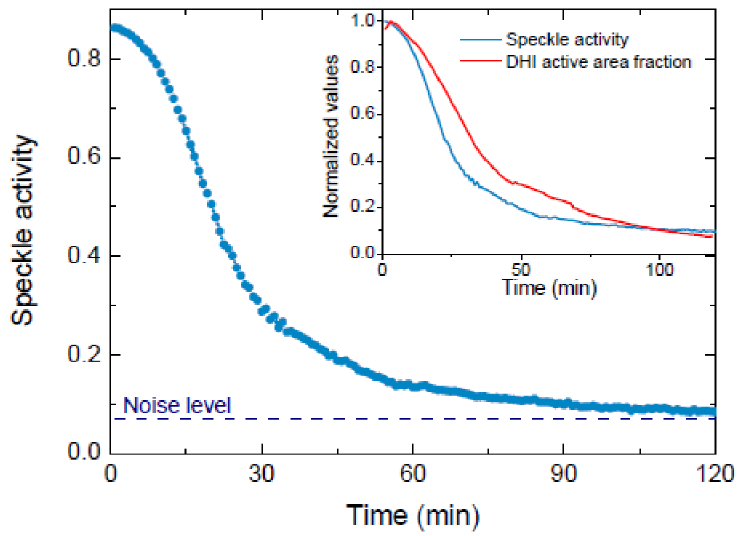

Fig. 6. Evolution of speckle activity of the drying enamel onto a vertical surface. The inset shows a comparison between the (normalized) curves obtained from dynamic speckle and DHI measurements.

To make a first estimation of the drying time one can exploit the nearly linear decaying segment of the curve.By extrapolating this line to the horizontal axis one could get an idea of how long will the paint take to dry**.** To perform this operation systematically and in real-time, i.e. as the data points of the curve are obtained, wesimulated a real-time acquisition of the holographic and speckle images and a real-time fitting of a sigmoidalcurve to the calculated data points.
The model used for fitting was based on the mentioned decaying sigmoidal curve, with the form:

$$f(t) = \frac{M-m}{1+\left(\frac{t}{t_0}\right)^p} + m \tag{4}$$

where M and m are the maximum starting value and the minimum value of the function, respectively, t is the time, t_0 is the time at which the function decays to the midpoint value between M and m and p (> 0) is the exponent related to the decaying rate. The relevant parameters for our estimation are t_0 and the slope of the curve at that point. The characteristic time t_0 is directly extracted from the fitting and the slope at that point is given by:

$$\left.\frac{df(t)}{dt}\right|_{t=0} = -\frac{p(M-m)}{4t_0} \tag{5}$$

With this information, the drying time can be easily estimated once the fitting function is adjusted to datapoints as they are obtained.
In Fig. 7 we compare the estimated drying time predictions from the curves of SA and active area fraction in DHI, respectively. We recall that the active area obtained from DHI interferograms requires a higher amount of numerical processing (image filtering and cropping, unwrapping, smoothing, and so on) than dynamicspeckle images. As it can be seen, during the first stages of drying, the estimated drying time obtained from SA data present more dispersion than those from DHI but converge quite fast (during the first 5 min) to the final value about 48 min. As a counterpart, DHI overestimates considerably the final value and it requires higher amount of experimental data (or time, ~ 12 min) to converge to the final value of about 65 min. The difference betweenboth estimated times is due to the shape of the sigmoidal function. The DHI active area fraction curve is clearly wider than that of SA (see Fig. 6) resulting in a higher estimated time.
In order to achieve a fast prediction of drying time, following the method proposed here, it is necessary to have sufficiently smooth curves. Otherwise the fitting procedure will be more likely to fail during the acquisition of the first data points. As it has been said before, the drying time estimated with the methods proposed here corresponds to the time after which the paint will reach a sufficiently steady surface state as long as the optical techniques can detect. But in general, as it is evidenced by the curves shown, one could ensure that the surface dry state will be achieved after the curves reach an asymptotic behavior. This will be

after an elapsed time of about twice the drying time estimated here.
As a second experience we observed the drying process of an enamel droplet deposited onto a horizontal surface. For this, we modified the DHI experimental setup as explained in the Experimental Section. We acquired holograms at 15 s intervals and speckle images at 1 s intervals. In Fig. 8(a) we show some processed interferograms of the droplet by comparing holograms at 15, 30 and 60 s intervals. Obviously, while reducing the analysis interval in DHI the resolution to detect smaller changes increases. In Fig. 8(b) we present the curves corresponding to the active area fraction calculated at those intervals.

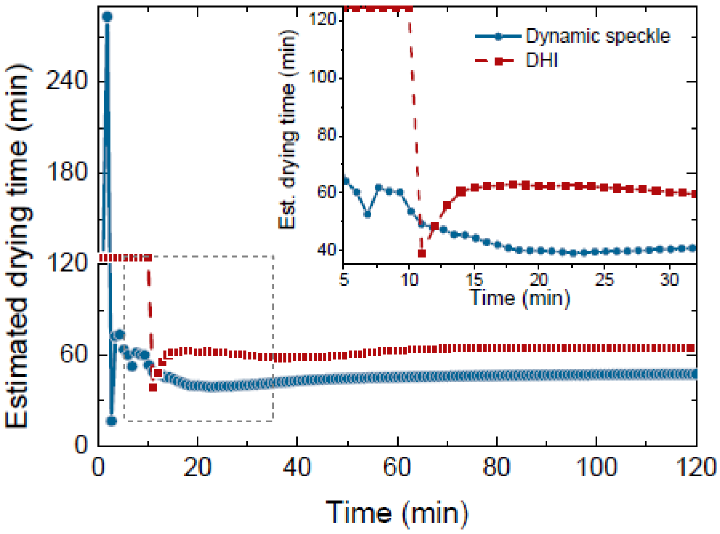

Fig.7. Predicted drying times by data fitting obtained from dynamic speckle and DHI methods of observation. In the corner is shows an applied region of the time interval where curves converge to their final values.

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The curves show that predictions of drying time will differ for the different observation intervals. This is reasonable since, as the paint dries, changes are progressively smaller and the resolution attainable at short observation times is not enough to detect them. However, at longer observation intervals the changes in the droplet surface become more evident in the interferograms.
During the first stages of drying the active area fraction curves are too noisy to make real-time predictions of drying time. Despite this, the time prediction is possible as soon as the curves start to decay by linearly extrapolating them to the horizontal axis as before. From this, we can conclude that DHI is useful to inspect advanced stages of drying paint provided the observation interval is suitably chosen. For example, the curve obtained at intervals of 60 s allows predicting the drying time after 90 min of observation. Although this observation period is quite long, the predicted time (~ 135 min) will be approximately the time necessary to achieve the surface dry state of the paint (in general greater than 3 hours). This supports the conclusion regarding the usefulness of DHI for analyzing the final stages of dryingpaint.

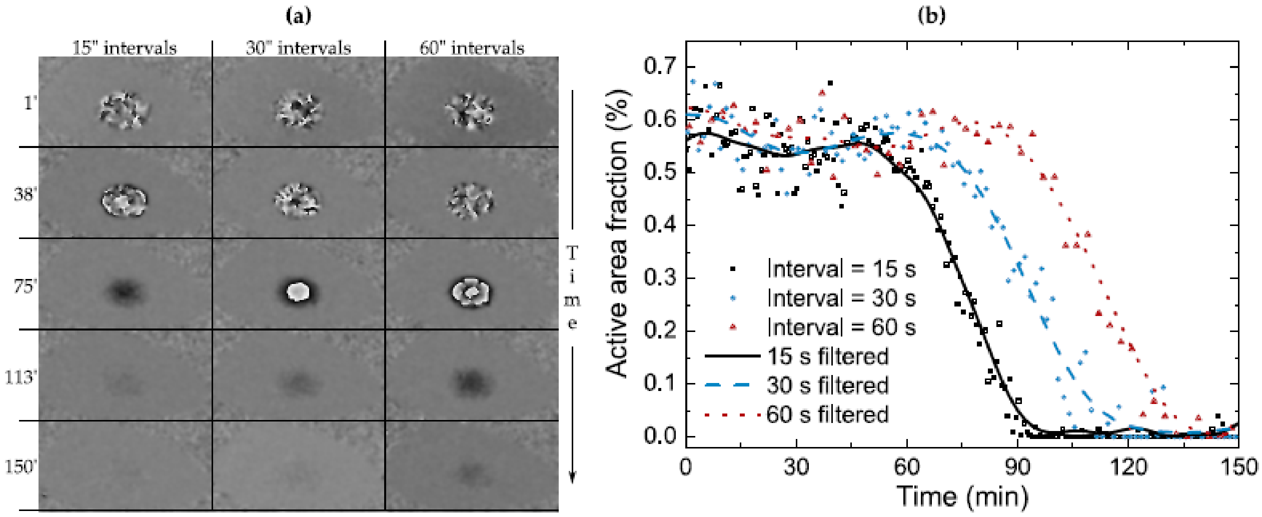

Fig. 8. Evolution of (a) a selection of processed interferograms and (b) active area fraction values, calculated at different intervals (15, 30 and 60 s).

For dynamic speckle measurements of the droplet we acquired images at 1 s intervals and the SA calculations were performed by taking N = 10 frames, which gives Δτ = 10 s. As before, a typical decaying curve was obtained, as seen in Fig. 9. The curve is quite smooth and starts to decay linearly from the beginning. If this happens, the fitting gets simpler since it is enough to perform a linear regression and extrapolate. For this, it is necessary to determine correctly the noise level (~ 0:3 in this case) due to the fact that extrapolation will strongly depend on this value. The estimated drying time can be readily approximated after about 10 min of observation and resulted in about 80 min. This time is greater than that of the first experience since the paint was distributed in a different way and dried in a static way, i.e. without flowing.

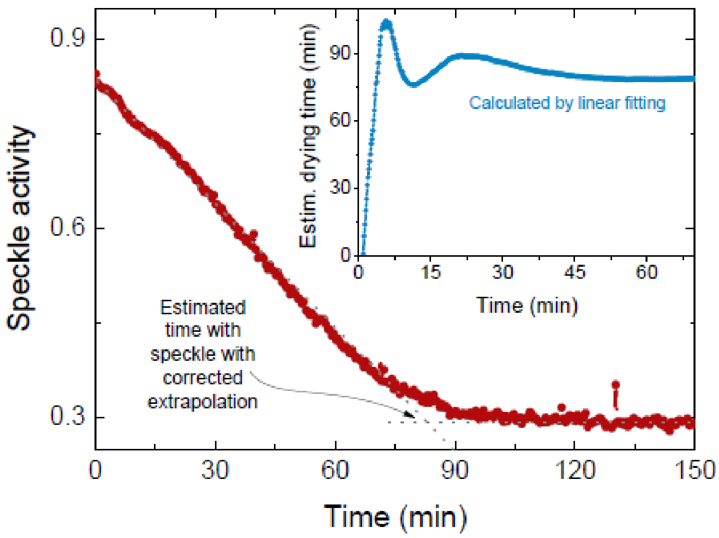

Fig. 9. Evolution of SA with time for the paint droplet. The curve follows a typical decreasing trend and starts to decay linearly, which simplifies the fitting procedure and the drying time estimation. The inset shows the estimated time through linear fitting of the SA curve.

To conclude, we have analyzed the possibility of estimating drying time (as defined above) by means of SA and DHI measurements. We found that, in fact, it is possible to estimate the drying time provided observations develop during a reasonable period of time, allowing to fit an adequate function (sigmoidal or linear) to theexperimental data. It is important to take into account that, in general, experiments to determine drying timeof paints require measurement periods in the order of a few hours. Therefore, we have set the basis to develop anestimation method that would allow to considerably reduce these times. Further work will be performed in orderto get to a standardized method with an adequate estimation of drying times and their corresponding errors andconfidence levels.

5 Conclusion

In summary, we have analyzed the possibility of estimating drying time of paints by implementing together dynamic speckle and holographic techniques. We showed that the time evolution of the dynamic speckle activity provides a quite simple way to achieve this goal after only a few minutes of observation. In turn, holographic interferometry could also serve for drying time estimation, but at the expense of a more complicated experimental setup, longer observation times and noise reduction algorithms. We have used simple algorithms for image processing and data fitting, which can be easily implemented for real-time measurements. These results conform a starting point for developing a suitable standardized method for determining drying time of paints and also other characteristics that might be relevant for paint manufacturers and users.

6 Acknowledgments

This work was possible due to funding resources from SorbalokSA® Company and R+D projects from Universidad Tecnológica Nacional (UTN) and UDC Universidade Da Coruña (Spain), ANPCyT and CONICET institutions of Argentina.

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